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TRW No. 29279-6006-RU-00

FINAL TECHNICAL REPORT
27 JANUARY 1976 THROUGH 15 NOVEMBER 1979

LONG TERM STORAGE TEST OF TITANIUM MATERIAL WITH LIQUID FLUORINE PROPELLANT

15 DECEMBER 1979

by

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W.D. English
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JPL CONTRACT NO. 954450

Prepared for

THE JET PROPULSION LABORATORY
Pasadena, California 91103

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DEFENSE AND SPACE SYSTEMS GROUP
ONE SPACE PARK • REDONDO BEACH, CALIFORNIA

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FOREWORD

This final report is issued under JPL Contract No. 954450 and covers the work performed from 1 February 1976 through 15 November 1979. The work was sponsored by the Jet Propulsion Laboratory with Mr. Louis Toth serving as the JPL Technical Manager. The program was performed as a joint effort of the Chemistry and Chemical Engineering Laboratory and the Materials Engineering Department.

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ABSTRACT

The compatibility of 6AL-4V Ti with propellant-grade GF_2 and LF_2 at 77 K for up to 3 years was investigated. Ti double coupons, annealed or heat treated, with 16 or 64 RMS finishes, were immersed in F_2 in individual Pyrex capsules and stored under LN_2 for 29 and 39 months. Pre-and post-immersion tests were performed on the propellant and coupons. Chemical analysis of the propellant did not reveal any significant changes due to titanium corrosion. Gravimetric, visual, microscopic, and metallurgical examination with pitting analysis did not reveal gross corrosion of the titanium although pitting appears to be greater after 39 months exposure. The increase in pit size and number raises the possibility of unpredictable crack propagation instability. Fracture toughness tests are necessary to define this possibility.

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GLOSSARY

In the context of this report, the following definitions will be applied.

Propellant

- **Sample:** A quantity of a fluid (gas, vapor, or liquid) withdrawn from a larger quantity of the same material, and assumed to be both representative and random.
- **Aliquot:** A sample whose quantity (volume or mass) is a known fraction of the total quantity from which it is withdrawn.

Titanium

- **Specimen:** A double-dogbone piece of titanium alloy, as furnished by JPL
- **Coupon:** A portion of titanium alloy cut mecahnically from the double-dogbone specimen.
- **Tensile Coupon:** A coupon of special configuration used for tensile testing.

Note that randomness of the specimens is not assured, and the coupons are definitely not random.

Procedures

- **Contact: / Immersion:** A compatibility experiment in which a specimen of titanium is exposed to F_2 under controlled conditions.
- **Test: / Characterization:** A determination of a property or composition of a specimen or sample of a material which will be or has been immersed in F_2

1. INTRODUCTION AND SUMMARY

There are many benefits to be gained from the use of liquid fluorine (LF_2) as the oxidizer for liquid propulsion systems aboard extended duration mission space vehicles. However, the design of appropriate propulsion systems utilizing LF_2 is hampered by inadequate critical information about the long term compatibility of the fluorine with materials of construction. The objective of the current program is to generate long term storage compatibility data for the titanium alloy 6AL-4V in contact with propellant grade liquid and vapor fluorine at a temperature of 77 K for 2- and 3-year periods. The program is oriented to develop data suitable for use in design of components which are in static or low-flow-rate contact with LF_2, such as tanks and some plumbing lines. The data are not applicable to highly stressed components, to components which are exposed to general or local rapid flow such as filter fibers and injectors, nor to frequently actuated dynamically loaded components such as valve seats or bellows.

The procedures utilized were developed previously for JPL Contract No. 953486, as reported in "Compatibility Testing of Spacecraft Materials and Space Storable Liquid Propellant," Final Report TRW No. 21362-6023-RU-00, May 1974.

Specimens of titanium 6AL-4V in two heat treat conditions (annealed, 147 KSI, and heat-treated, 175 KSI) and two surface finishes (16 and 64 RMS) were supplied by JPL. Selected specimens were: a) kept unexposed; b) exposed to GF_2 for 1 hour (passivated); c) immersed in LF_2 and GF_2 for 29 months; and d) immersed in LF_2 and GF_2 for 39 months. Control specimens of LF_2 were stored for 29 and 39 months. The fluorine was analyzed before the immersion, and both control and immersion samples were analyzed after the immersion. The metal specimens were characterized after each of the four modes. Characterization included weight change, tensile properties measurement, visual examination and corrosion mapping, microscopic examination, metallography, including pit development evaluation, and scanning electron microscopy (SEM).

There was no significant change in the composition of the fluorine in contact with titanium. However, when the gas was left to stand in contact

with 18-8 CRES at room temperature, it became contaminated with a volatile chromium fluoride and became darker in color. The contaminant was unavoidably transferred into the test capsules.

There were minor weight gains for specimens exposed in the 29- and 39-month tests. Tensile properties did not change for either vapor- or liquid immersed specimens. No significant exposure effects were found by the corrosion mapping, visual, microscopic, or SEM inspections. However, the pitting analysis revealed an accelerating attack rate which had two separate characteristics. The number of pits and their area increased markedly, with a greater increase in the 29-39 month period than in the 0-29 month period, however the depth of most of the pits remained constant, except for the 64 RMS coupons, especially those in the annealed condition, which after 39 months showed appreciable formation of deeper pits, about 5 times as deep as the average for all coupons.

There is a possibility that the increase in pitting severity with time may eventually lead to catastrophic failure of the alloy by crack instability. This failure mode is dependent on both the fracture toughness of the alloy and the applied stress.

2. TECHNICAL DISCUSSION

2.1 OBJECTIVE

The objective of this program is to generate and evaluate data on the effects from long-term contact for up to three years of titanium alloy 6AL-4V with liquid and vapor propellant-grade fluorine at a temperature of 77 K. The purpose is to supply missing critical data for the design of tankage for extended duration mission space vehicles using fluorine as oxidizer.

2.2 APPROACH

The approach used in this program is:

- Characterize the initial condition of the titanium alloy.
- Characterize the titanium alloy after passivation with GF_2.
- Characterize the initial condition of the fluorine.
- Contact individual specimens of the titanium with individual quantities of liquid fluorine for selected durations of up to 3 years at specified test conditions.
- Store control samples of fluorine without titanium under identical conditions for the same time intervals.
- Separate the fluorine from the titanium at the end of the contact period.
- Characterize the post-contact condition of the titanium alloy specimens.
- Characterize the post-contact condition of the fluorine.
- Characterize the control samples of fluorine.
- Characterize any new substances which were formed during the contact period.
- Calculate any changes which occurred in the titanium, the fluorine, and the test system.
- Evaluate the changes as functions of test duration and test conditions.
- Draw conclusions about the significance of the results and make appropriate recommendations relative to utilization of titanium in contact with fluorine and also for additional studies which may be necessary.

2.3 METHOD

2.3.1 Introduction

The experimental portion of the program consists of three parts:

1. Characterization of the propellant fluorine.
2. Characterization of the titanium alloy specimens.
3. Immersion of the alloy specimens in fluorine.

Characterization of the fluorine and of the titanium is done before and after the immersion. Controls are included in which the immersion tests are simulated, but without contact between the two materials.

The program is arranged in five sequential tasks:

Task 1. Install equipment. Conduct pre-immersion characterization of specimens and propellant.

Task 2. Initiate immersion.

Task 3. Conduct immersion.

Task 4. Terminate 2-year immersion. Conduct post-immersion characterization.

Task 5. Terminate 3-year immersion. Conduct post-immersion characterization.

This report is the final technical report on the program. In it we present all the procedures, observations, results, analyses, and conclusions for the complete program. Interim technical reports which have been published during the program include:

- "Program Plan and Schedule," TRW No. 29279-6001-RU-00, 10 February, 1976.
- "Informal Progress Report, 28 January 1976 through 21 May 1976," TRW No. 29279-6002-RU-00, 21 May 1976.
- "Final Letter Report No. 2, 28 January 1976 through 27 July 1976," TRW No. 29279-6003-RU-00, 27 July 1976.
- "Final Letter Report No. 3, 27 July 1976 through 5 July 1978," TRW No. 29279-6004-RU-00, 5 July 1978.
- TRW Operating Procedure, "Removal of Fluorine and Analysis of Fluorine in Long Term Storage TEsts of Titanium Specimens with Liquid Fluorine" No. YS-28T-15, 18 October 1978.

- "Final Letter Report No. 5, 6 July 1978 through 1 April 1979," TRW No. 29279-6005-RU-00, 25 April 1979.

2.3.2 Plan

The original plan is described in this section. The actual operations which were conducted and the procedures used are described in detail in Section 2.3.4.

2.3.2.1 Materials

Titanium specimens, 36 each, in selected metellurgical and surface finish conditions, were to be furnished by JPL (see Section 2.3.3.1). Propellant grade fluorine and the apparatus items were to be procured or supplied by TRW.

2.3.2.2 Pre-Immersion Characterization of 6AL-4V Titanium Alloy Specimens

All 36 specimens were to be weighed on an analytical balance, accurate to $\pm 1 \times 10^{-4}$ g, immediately upon removal from their protective containers, and the weights recorded.

Preimmersion characterizations of the 6AL-4V titanium alloy were to be performed on eight, and mechanical tests on four, for a total of twelve specimens. The preimmersion surface condition analysis was to include stereo, light optical, and scanning electron microscope examinations. Pitting analyses by Champion's method (Reference 1) were to include the measurement of pit concentrations, sizes, and depths. Table I is a matrix of the tests which were planned. In order to minimize effects of exposure to the humid atmosphere, all operations in the surface examination were to be conducted under purge by dry argon, in a dry argon atmosphere, or in vacuum. Immediately after the examination of each specimen was completed, it was to be sealed in an inert atmosphere.

The mechanical property tests -- ultimate tensile strength, yield strength, and percent elongation -- were to be conducted using an Instron Universal Test Machine for the measurement. Because two tensile coupons are made from each double dogbone specimen, a total of 8 individual tensile tests were planned. Following the tensile tests, the coupons were to be resealed in an argon atmosphere.

Table I. 6AL-4V Titanium Specimens - Pre-immersion Characterization

Item	Number of Specimens			
Material condition	Heat Treated		Annealed	
Specimen Surface Finish (micro inches)	16 RMS	64 RMS	16 RMS	64 RMS
1.0 Examination and Metallurgy				
1.1 Sealed Controls (Cleaned: no passivation)	1	1	1	1
1.2 Sealed Controls (Cleaned; passivated)	1	1	1	1
2.0 Mechanical Properties Tests				
2.1 Room Temperature (298 K) (Cleaned; passivated)	1		1	
2.2 LN_2 Temperature (77 K) (Cleaned; passivated)	1		1	
Totals	4	2	4	2

2.3.2.3 Fluorine Characterization

Prior to use, a complete assay for the fluorine was to be performed perspecification MIL-P-27405, "Fluorine Propellant, Type II, Liquid." The sample analyzed was to be drawn from the batch to be used in this program. Results of this analysis are described below, in Section 2.3.3.2.

2.3.2.4 Liquid Fluorine Controls

Eight each 10 ml volumes of liquid fluorine were to be loaded into specimen capsules and stored as control samples in the Cryoflask storage unit, as summarized in Table II, together with the titanium specimen compatibility tests for the same durations.

2.3.2.5 Preparation of Specimens for Storage

A total of twenty-two 6AL-4V specimens were to be placed in cleaned glass capsules and sufficient liquid fluorine added to cover the lower coupon of the double dog-bone specimens. These specimens were to be prepared ready for storage at 77 K.. The assignment of specimen conditions and finishes was to be that shown in Table III.

Table II. LF_2 Storage Tests at 77 K

Test Group	Storage Period, Months	Number of Capsules
A	24	4
B	36	4
		Total 8

Table III. 6AL-4V Titanium Specimens Storage in LF_2 at 77 K

Item	Test Group	Number of Specimens and Capsules			
Material condition		Heat Treated		Annealed	
Specimen Surface Finish (micro inches)		16 RMS	64 RMS	16 RMS	64 RMS
Storage Period (months)					
24	A	3	3	2	2
36	B	4	4	2	2
Totals		7	7	4	4

The assignments in Table I and III total 34 specimens. The two additional specimens supplied by JPL were to be for spares and replacements. Assignment of individual specimens to particular tests (passivation, 24-month, or 36-month) was to be arbitrary, with no requirement for randomization.

2.3.2.6 Immersion Capsule Storage at 77 K

All loaded capsules were to be transferred to carrying cannisters in the Cryoflask cryogenic storage unit, and a chart to be prepared showing the location of each capsule as it was emplaced. The cryogenic storage unit was to be transferred to a barricaded storage facility with a LN_2 supply. The LN_2 in the storage unit was to be replenished at least 2 times

per week. Storage was to be at 77 ± 4 K, at an internal pressure of 300 ± 160 torr, the vapor pressure of F_2 in the temperature range.

A table assigning each titanium and each fluorine control specimen to one of the 24 month or 36 month storage periods was to be prepared.

2.3.2.7 Immersion Termination

At the end of 24 months for Group A and of 36 months for Group B, the capsules were to be removed from storage. It was planned to open them, remove the fluorine by evaporation, sample and analyze the fluorine from 1 capsule of each condition of the titanium specimens (4 total), and also to analyze the F_2 from the control tests.

2.3.2.8 Specimen Post-Immersion Testing

It was planned to weigh each specimen and to conduct visual, stereo, and magnified examinations, SEM, metallurgical analysis, pitting evaluation, and mechanical property tests on selected specimens. Tables IV and V outline the post-immersion tests for Group A and Group B respectively. These test plans were designed after the immersions had been initiated and their increased level of detail, compared to Table I, reflects the experience gained in the program.

2.3.3 Materials and Apparatus

2.3.3.1 Titanium

Ti 6AL-4V in the form of specimens conforming to JPL Drawing 10056227, Revision C, entitled "Metallic Specimen, Double Dogbone, P/N 10056227-1" were furnished by JPL (Figure 1). All specimens have a Vee notch at one end to indicate the end for vapor exposure during immersion tests, and all are engraved with a number code at both ends of one side. The specimens were provided by JPL in a variety of surface finish and heat treat conditions, as listed in Table VI.

All specimens were cleaned by JPL in accordance with JPL Specification ES504574, Revision C, entitled, "General Cleaning Requirements for Spacecraft Propulsion Systems," and packaged to prevent recontamination. This cleaning procedure prohibits use of halogenated and fluorinated solvents, and permits only isopropyl alcohol as an organic solvent.

Table IV. Group A Post-Immersion Tests

2 Year Immersion

10 Specimens in Immersion - 20 Coupons for Testing - 4 F_2 Controls

Specimen Type / Test Function	Number of Tests				
	Heat Treated		Annealed		
	16 RMS	64 RMS	16 RMS	64 RMS	F_2 Controls
Weigh/Specimens	3	3	2	2	--
Examination and Mapping/ Complete Specimens	3	3	2	2	--
SEM/Coupons: Vapor	1	1	1	1	--
Liquid	1	1	1	1	--
Metallography/Coupons: Vapor	2	2	2	2	--
Liquid	2	2	2	2	--
Pitting/Coupons: Vapor	1	1	1	1	--
Liquid	1	1	1	1	--
Chemical Analysis of F_2/Capsules	1	1	1	1	2
Tensile Tests/Coupons:					
Vapor, 298	2	2	1	1	--
Vapor, 77	1	--	1	--	--
Liquid, 298	2	2	1	1	--
Liquid, 77	1	--	1	--	--

Table V. Group B Post-Immersion Tests

3 Year Immersion

11 Specimens in Immersion - 22 Coupons for Testing - 4 F_2 Controls

Specimen Type / Test Function	Number of Tests				
	Heat Treated		Annealed		
	16 RMS	64 RMS	16 RMS	64 RMS	F_2 Controls
Weigh/Specimens	3	4	2	2	--
Examination and Mapping/ Complete Specimens	3	4	2	2	--
SEM/Coupons: Vapor	1	1	1	1	--
Liquid	1	1	1	1	--
Metallography/Coupons: Vapor	2	2	2	2	--
Liquid	2	2	2	2	--
Pitting/Coupons: Vapor	1	1	1	1	--
Liquid	1	1	1	1	--
Chemical Analysis of F_2/Capsules	1	1	1	1	4
Tensile Tests/Coupons:					
Vapor, 298	2	3	2	2	--
Vapor, 77	1	1	--	--	--
Liquid, 298	2	3	2	2	--
Liquid, 77	1	1	--	--	--

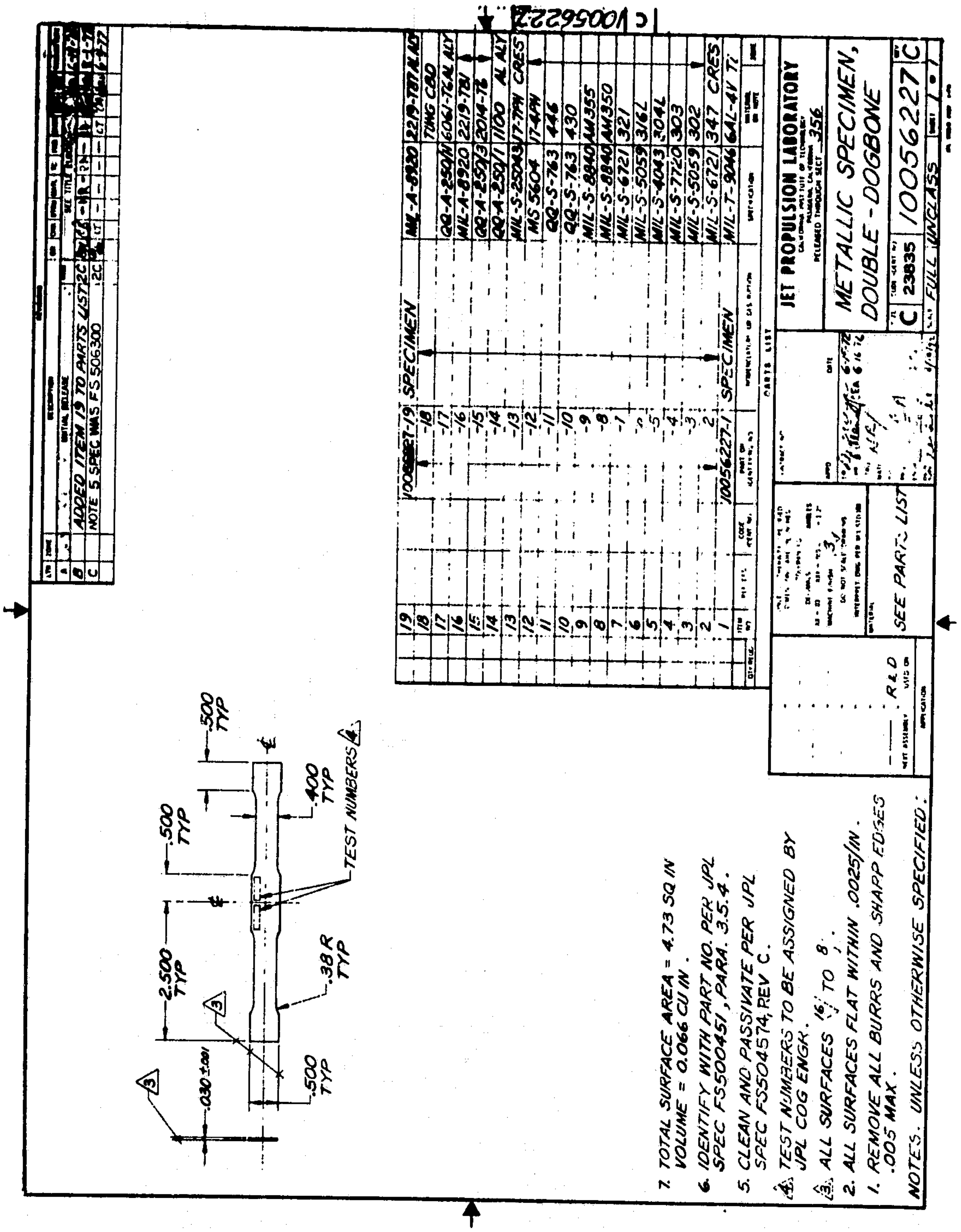

Figure 1. Metallic Specimen, Double Dogbone

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Table VI. Titanium Specimen Condition Distribution

Heat Treat Condition and UTS / Surface Finish, μ in.	Specimen Categories			
	Heat Treated 175 KSI		Annealed 147 KSI	
	Quantity	Code Nos.	Quantity	Code Nos.
16 RMS	12	7500-11	8	7550-57
64 RMS	10	7520-29	6	7570-75

Documentation of the alloy used in fabrication of the specimens is given in Appendix A. Specifications for preparing the particular heat treat conditions and surface finishes, and for tests to establish that the conditions and finishes conform to specification are given in Appendix B.

2.3.3.2 Fluorine

Gas fluorine was obtained in a size A cylinder from Air Products, purchased per Specification MIL-P-27405, Fluorine Propellant. It was used "as received", and was NOT passed through a NaF tower. Portions of the material were withdrawn from the cylinder into an evacuated transfer system. Portions as required were analyzed according to the procedure for "Type II, Liquid" in the above specification. An additional portion of the fluorine was introduced, by means of the vacuum transfer system, into an evacuated 10-cm infrared spectrometer cell equipped with silver chloride windows. The fluorine pressure in the cell was 760 torr. A spectrum was run between 850 cm^{-1} and 4000 cm^{-1} and the absorbancies at 1280 cm^{-1} for carbon tetrafluoride and at 1050 cm^{-1} for silicon tetrafluoride were compared with standard curves for these to compound to determine the concentrations. The results are presented in Table VII. Because the propellant composition affected operations for transferring F_2, it is discussed in the following paragraphs instead of in the Program Results Section.

Unidentified non-condensable species make up at least 0.1% v/v. Such species may include oxygen, nitrogen, helium, and/or carbon tetrafluoride. Table VIII shows the volatility of a number of possible impurities in the fluorine, in terms of individual boiling points.

Non-condensables can cause problems during vacuum transfer operations at cryogenic temperatures. Helium is particularly bothersome during low pressure cryompumped transfer from a liquid supply at 77 K to a receiver at 4.3 K. The helium does not condense at the low pressure inside the

Table VII. Analysis of Starting Fluorine

Constituent	Specification Limit	Analysis Results
Fluorine Assy, % v/v	99.0 (min)	99.21
Sum of Hydrogen Fluoride + Carbon Dioxide, % v/v	0.1 (max)	0.04
Non-Condensables, % v/v	0.9 (max)	0.28
Carbon Tetrafluoride, % v/v	NR*	<0.1
Silicon Tetrafluoride, % v/v	NR*	<0.1

*NR - Not required

Concentration % v/v, refers to the vapor phase composition when all materials have volatized without fractionation. It is equal to % mol/mol.

Table VIII. Boiling Points of Possible Impurities in Fluorine

Species	B.P. (K)
He	4.3
N_2	77
CO	82
F_2	85
O_2	90
OF_2	128
CF_4	145
SiF_4	178 (sublimes)
CO_2	195 (sublimes)
HF	299

manifold and its vapor causes development of a back-pressure in the receiver, slowing and ultimately preventing transfer of fluorine. The observed behavior of the fluorine during transfers for this program indicated that helium was present as a significant portion of the non-condensable fraction, but that it was not the only such material present. To prevent the back pressure problem, it was necessary to subject the liquid fluorine to a dynamic vacuum prior to and periodically during its transfer into the test capsules. This obviously changed the composition of the fluorine in which the titanium was immersed.

2.3.3.3 Apparatus

The apparatus used for installing and conducting the immersion tests will be described as three items. These are the compatibility capsule, the fluorine transfer manifolds, and the cryogenic storage facility.

a) Compatibility Capsule

The capsules were manufactured by TRW from borosilicate glass tubing. Their design is sketched in Figure 2. The capsules were fabricated in two sections for later sealing at the fusion joint. Annealing of both sections was accomplished by heating in a furnace at 833 K and holding at this temperature for 0.25 hour. The heating rate was not considered critical, and the cooling to ambient was at the normal cooling rate of the furnace. After the capsules were manufactured and annealed, they were cleaned, in a clean room, in an ultrasonic bath with an Alconox solution, washed with deionized water and dried in a vacuum oven at 383 K, and packaged to prevent recontamination.

The two sections were joined after a titanium specimen was placed in the lower section, using a portable hydrogen-oxygen torch, with a purge flow of argon through the capsule to protect the specimen. This process is described in Section 2.3.4.1(a).

b) Fluorine Transfer Manifolds

The manifold used for filling the capsules with fluorine is sketched in Figure 3. This manifold was used for passivation of specimens with gas fluorine, and to load liquid fluorine into capsules, both liquid fluorine controls and those containing specimens.

A fluorine cylinder was connected directly to the manifold with 6 meters of 0.6 cm O.D. copper tubing. The manifold was constructed of 0.6 cm O.D. stainless steel tubing and the attached valves were Hoke No. 4251N6Y fabricated from stainless steel. Swagelok and AN flare connections were used. Copper tubing was

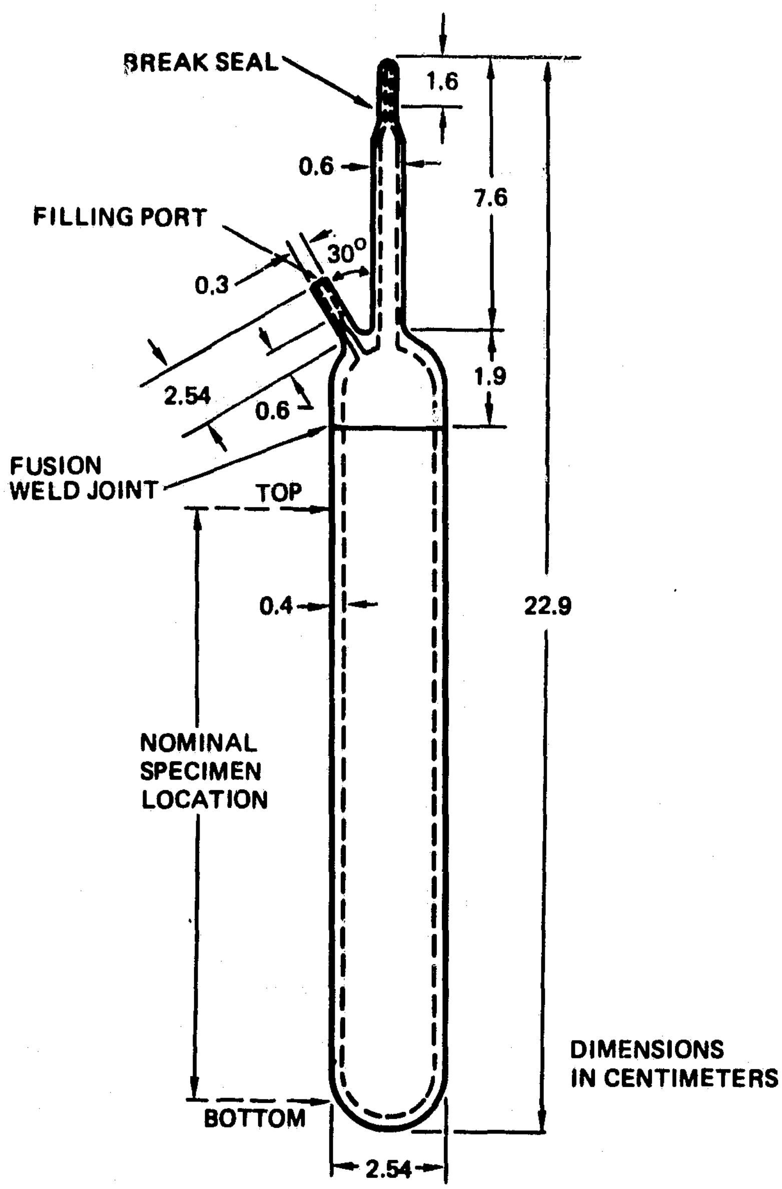

Figure 2. Compatibility Capsule

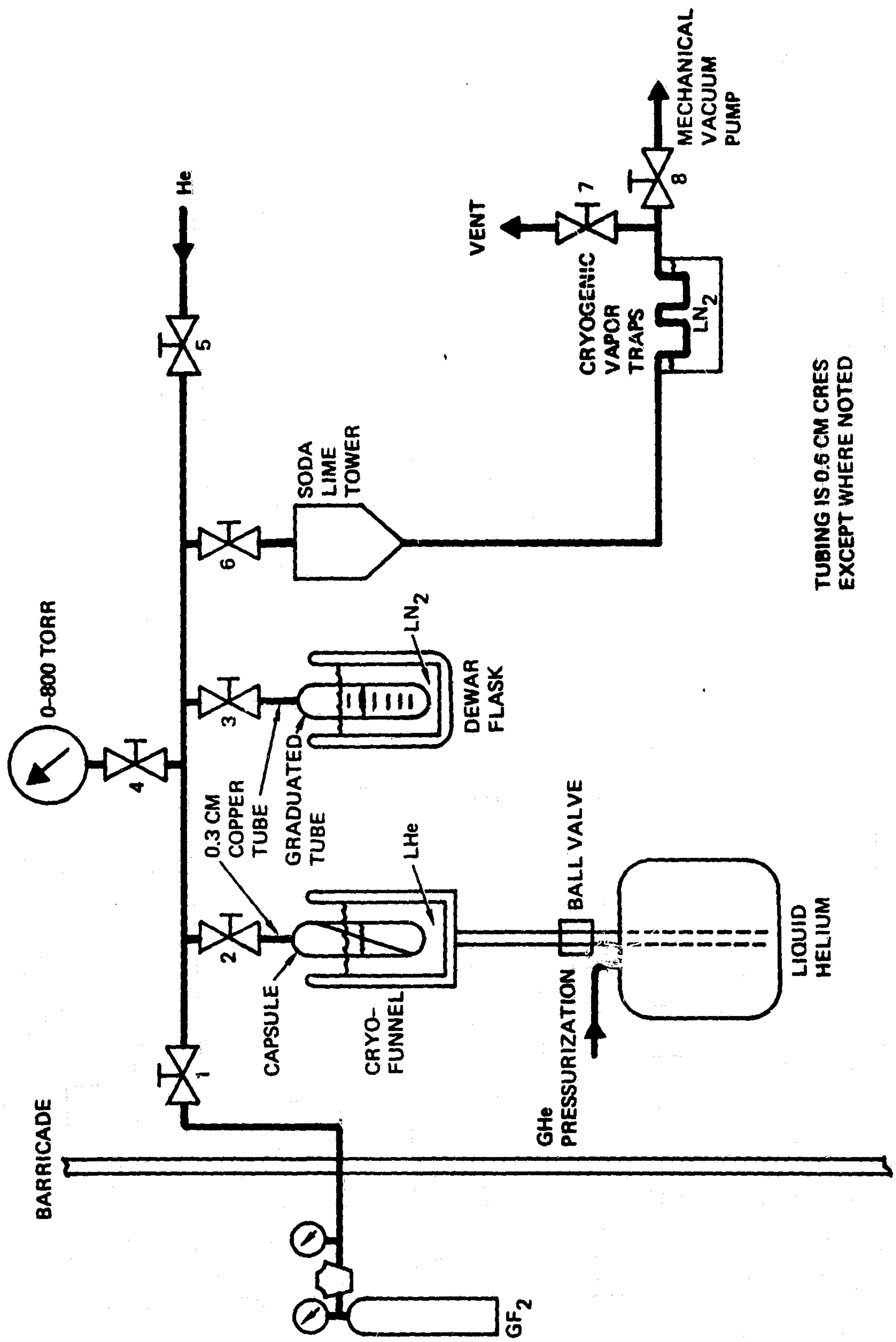

Figure 3. Fluorine Supply Manifold

used to connect the manifold to the glass capsule and to the graduated glass volume tube; the copper lines were attached to the capsule and the volume tube by means of Swagelok fittings with Teflon ferrules. Liquid helium was maintained around the sample capsule by use of a Cryofunnel. The Cryofunnel was simply a jacketed tube, 5 cm I.D. and 25 cm long. This jacketed tube was connected to a 1.25 cm O.D. stainless steel tube which was inserted into a liquid helium storage flask Dewar through a 1.25 cm ball valve. Below the ball valve was a 0.3 cm O.D. valved line for pressurizing the storage flask with 2-6 psig helium. To raise liquid helium into the Cryofunnel, the storage flask was pressurized and the pressure maintained until the liquid helium rose to the desired level. After the operations with the capsule were completed, the liquid helium was returned by placing a cover on the Cryofunnel, and the vapor pressure of helium forced the liquid to return to the storage Dewar.

Copper tubes, 0.6 to 1-meter long and 0.3 cm O.D., were attached at one end to Valves 2 and 3. The tube at Valve 3 was attached at the other end to a heavy walled graduated glass tube, and the tube at Valve 2 was attached to the fill port of a capsule. To remove absorbed gases and moisture from the samples and inside capsule walls, Valves 2 and 3 were opened and the manifold was evacuated, and the entire system held at a pressure below 0.1 torr for one hour while being pumped. Liquid nitrogen was maintained around the cryogenic vapor traps upstream of the vacuum pump. Before filling the capsule with liquid fluorine or for preparation of passivated-only specimens, the capsules and contents were passivated with fluorine by introducing one atmosphere of fluorine and permitting it to remain for one hour. The capsules were then evacuated to less than 0.1 torr and held under this condition for one hour, before proceeding to the appropriate subsequent step in the procedure.

The manifold used for removal of fluorine after completion of the immersion test is sketched in Figure 4. This is actually just a segment of a larger, multipurpose fluorine manifold. The capsule to be emptied is attached to the ball valve seal breaker by use of a Swagelok fitting with Teflon ferrules. The system is passivated carefully prior to attachment of a capsule to be emptied of fluorine.

c) Cryogenic Storage Facility

A Cryo-flask was used for the storage of the specimens and liquid fluorine. The Cryo-flask, manufactured by Minnesota Valley Engineering Company, has the configuration shown in Figure 5. This device is thermally efficient; after filling with LN_2 it will contain 25 percent of the initial fill after four weeks storage with no topping off. It was filled with liquid nitrogen and then cannisters containing up to four capsules each were placed in the Cryo-flask. Twice each week the Cryo-flask was replenished with liquid nitrogen. The capsules remained in storage until removed according to the schedule.

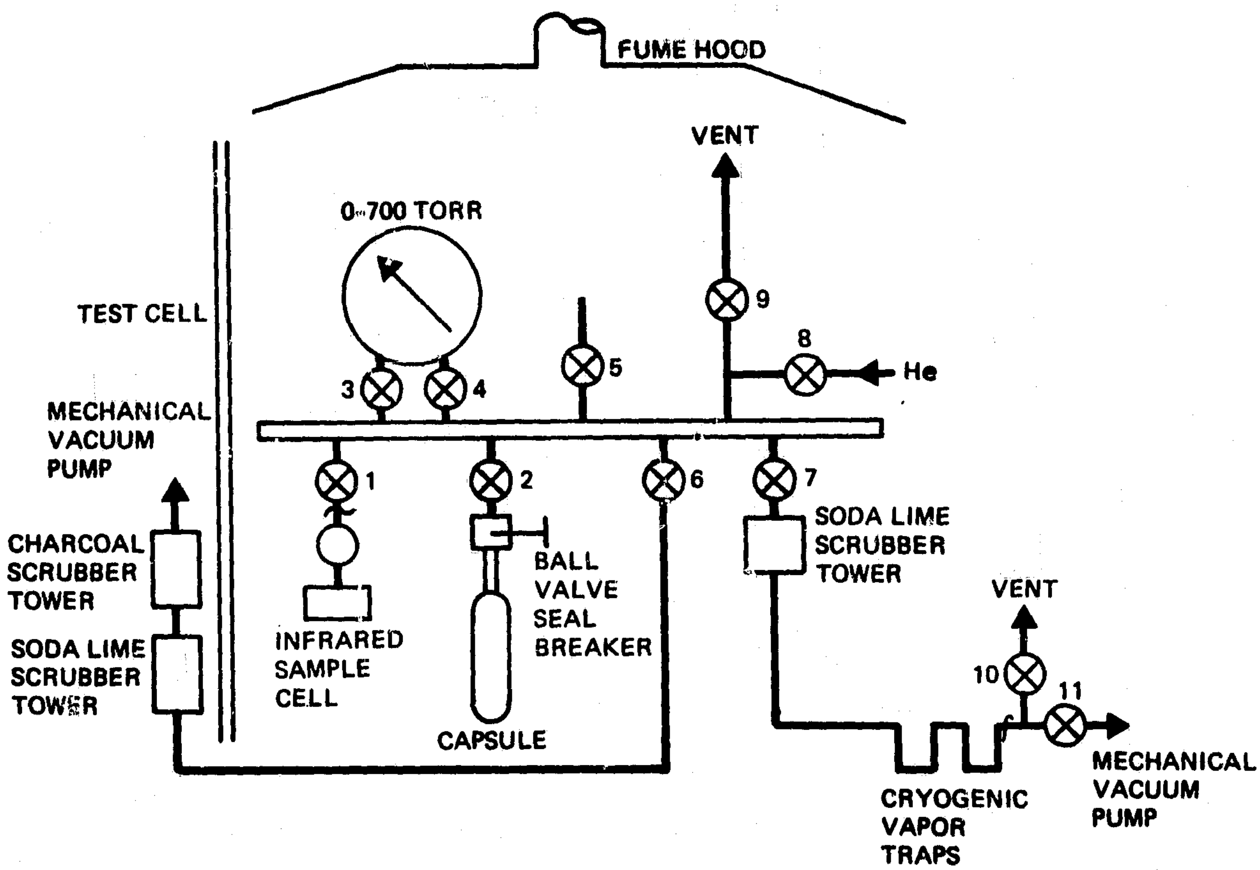

Figure 4. Fluorine Removal and Analysis Manifold

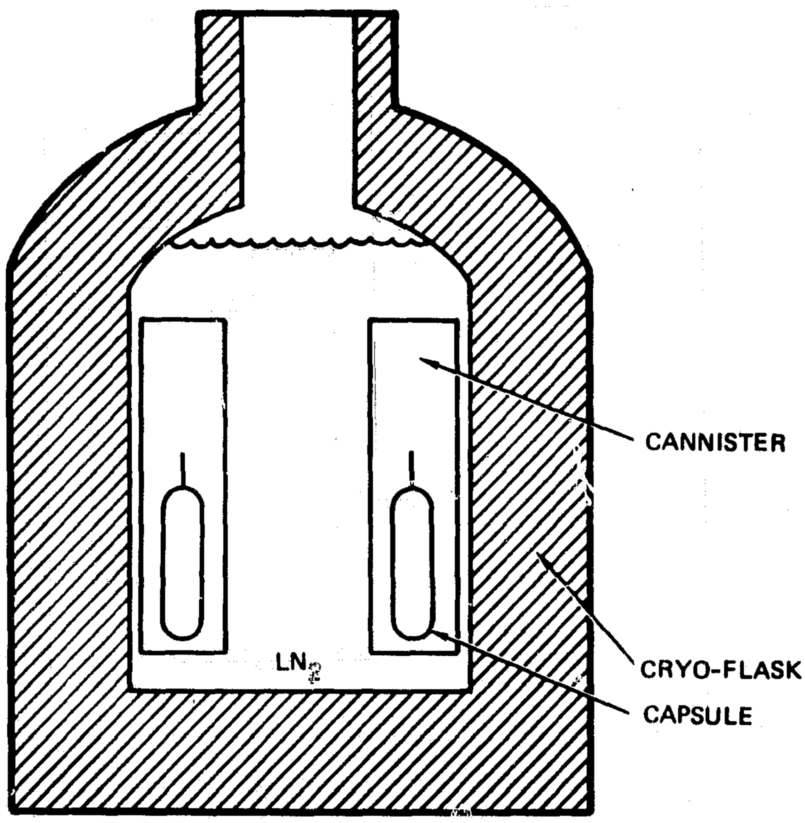

Figure 5. Cryoflask with Specimen Capsules in Cannisters

The apparatus used in characterizing the titanium specimens included the following:

> The tensile tests were conducted on an Instron Model TTCLM1-6 Universal Testing Machine. The strain rate was 0.50 inch/minute, and a 0.2% offset was used to determine the yield strength.
>
> All pit dimensions were measured on a Reichert Research Metallograph.
>
> SEM photographs were obtained using a JOEL Model JSM-2 scanning electron microscope.

2.3.4 Procedures

The actual operations carried out and identification of the specific test items utilized are described in this section. Attention is drawn to significant variances from the original plan. A chart showing the history of each specimen throughout the program is presented as Table IX. Table IX.

The most significant changes from the plan described in Section 2.3.2 are

- 29 month immersion for Group A - the extension, agreed to by the JPL Technical Manager, occurred as a result of some high priority technical problems on other programs.
- 39 month immersion for Group B
- Only 3 specimens of heat treated 16 RMS titanium in 36 months immersion.
- Post-immersion characterizations were done on many more specimens than originally planned.

Results are not given in this section except where the observations required a change in procedure, then summary data is presented to explain the change. Detailed results will be found in Section 2.3.5.

2.3.4.1 Fluorine Transfer Operations

(a) Passivation/Immersion Initiation

All specimens except 7508 and 7511 were sealed into capsules by the following technique. The work was conducted in a clean area to reduce introduction of contamination:

(i) The bottom half of a capsule was attached to a vertical support with a clamp. The top half of a capsule was attached by flexible tubing at the capsule fill port to a source of filtered argon for

Table IX. Specimen History Matrix

Specimen Identification	As Received State				Weight No. 1	Conditioning		Weight No. 2	Pre-Immersion Characterization						Immersion Test Duration			Weight No. 3	Post-Immersion Characterization					
	Heat Treated		Annealed						Tensile		Examination								Tensile		Examination			
	16 RMS	64 RMS	16 RMS	64 RMS		Passivated	Not Pass.		298 K	77 K	Visual	SEM	Metallographic	Pitting	None	29 Mo	39 Mo		298 K	77 K	Visual	SEM	Metallography	Pitting
LF_2 Control #1																	X							
LF_2 Control #2																	X							
LF_2 Control #3																	X							
LF_2 Control #4																	X							
LF_2 Control #5																X								
LF_2 Control #6																X								
LF_2 Control #7															Note 1									
LF_2 Control #8															Note 1									
7500	X				X		X	X	X		X	X	X	X	X									
7501	X				X	X		X			X	X	X	X	X									
7502	X				X	X		X	X X		X	X	X	X	X									
7503	X				X	X		X		X X	X	X	X	X	X									
7504	X				X	X											X	X	T	B	X	T B	T B	T B
7505	X				X	X										X		X	T B		X	T B	T B	T B
7506	X				X	X											X	X	B	T	X		T B	
7507	X				X	X											X	X	T B		X			
7508	X				X										Note 1									
7509	X				X	X										X		X	B	T (Note 3)	X			
7510	X				X	X										X		X	T	B	X		T B	T B
7511	X				X										(Note 1)									
7520		X			X		X	X	X		X	X	X	X	X									
7521		X			X	X		X			X	X	X	X	X									
7522																X		X	T B		X		T B	
7523		X			X	X											X	X	T	B	X	T B	T B	
7524		X			X	X											X	X	B	T	X		T B	T B
7525		X			X	X											X	X	T B		X			
7526		X			X	X											X	X	T B		X			
7527		X			X	X										X		X		T B	X	T B	T B	T B
7528		X			X	X										X		X	T B		X			
7529		X			X		X								Note 2									
7550			X		X		X	X	X		X	X	X	X	X									
7551			X		X	X		X			X	X	X	X	X									
7552			X		X	X		X	X X		X	X	X	X	X									
7553			X		X	X		X		X X	X	X	X	X	X									
7554			X		X	X											X	X	T B		X	T B	T B	
7555			X		X	X										X		X	B	T (Note 3)	X	T B	T B	
7556			X		X	X											X	X	T B		X		T B	T B
7557			X		X	X										X		X	T	B	X		T B	T B
7570				X	X		X	X	X		X	X	X	X	X									
7571				X	X	X		X			X	X	X	X	X									
7572				X	X	X										X		X		T B	X	T B	T B	T
7573				X	X	X											X	X	T B		X		T B	
7574				X	X	X											X	X	T B		X	T B	T B	T B
7575				X	X	X										X		X	T B		X		T B	T B

Note 1: Capsule broke during test initiation.
Note 2: Spare specimen, not tested.
Note 3: Coupon failed in grip region, no data.

T = Top coupon from specimen; vapor phase exposure.
B = Bottom coupon from specimen; liquid phase exposure.

purging. The operator wore white, lint-free nylon gloves and used forceps with Teflon-coated tips to place a specimen in the bottom half of the capsule. Then into each capsule with a specimen for liquid immersion, (thus omitting 7500-3, 7520-1, 7529, 7550-3, and 7570-1) were inserted two firepolished borosilicate glass rods, 0.7 cm diameter by 6.4 cm long. The glass rods, used to minimize the volume of liquid required, were placed one on each side of the specimen. The top half of the capsule, with a slow flow of purge argon flowing, was placed on the bottom half, and the two sections were fused together using hydrogen-oxygen portable glass blowing torch. The break-seal portion of the capsule was then formed. The argon flow was continued until the glass had cooled. In addition to the capsules which contained titanium specimens, eight empty capsules were fabricated for LF_2 controls. All completed capsules were inspected after cooling, using a polariscope to detect strains in the glass: None were found. The capsules were then enclosed and sealed in individual clean plastic bags before removal from the clean area and transport to the propellant laboratory.

Passivation of all capsules, except those containing specimens 7500, 7520, 7529, 7550, and 7570, was carried out as follows:

(ii) The filling manifold described in Section 2.3.2.3(b) and shown in Figure 3 was used. A capsule was attached to the manifold by copper tubing at the fill port. The system was evacuated to a pressure below 0.1 torr and held at that pressure for one hour. The vacuum pump was then valved off, and gas fluorine was admitted to the system to a pressure of one atmosphere, and allowed to stand for one hour. The fluorine was then evacuated from the system through the absorption tower, and the pressure reduced below 0.1 torr by a dynamic vacuum for at least one hour.

Non passivated control specimens 7500, 7520, 7550, and 7570, and spare specimen 7529 were handled as follows:

(iii) The same procedure as in (ii) was followed except that the capsules were pressurized for one hour with one atmosphere of helium in place of fluorine.

Capsules which were to be filled with liquid fluorine were not sealed off at this step -- further processing of these items is described at (v)

(iv) The passivation-only and unpassivated control capsules were then filled with GHe at one atmosphere and the fill port was fused closed while the capsule was still attached to the manifold; a hydrogen-oxygen portable torch was used.

For those capsules to be loaded with LF_2, 10 ml (15.5 g) of LF_2 at 77 K accurately locates the vapor-liquid interface midway between the two dogbone sections. The additional procedures are:

(v) The manifold was again evacuated. LN_2 was placed in the Dewar around the graduated glass tube, and gas fluorine was slowly introduced through Valve 1 to the manifold. When 10 ml of fluorine had condensed into the graduated glass tube, the fluorine supply was shut off at Valve 1. Liquid helium was introduced to the Cryofunnel (See Section 2.3.3.3(b)), and Valve 2 was opened and all the fluorine distilled from the graduated tube into the capsule where it froze. Figure 6 is a schematic of a loaded capsule.

Condensable gases with a slight vapor pressure and non-condensable gases were present in fluorine in sufficient quantities to cause problems when fluorine condensed at 77 K was cryopumped to a capsule at 4.3 K. Table VIII is a list of the boiling points of some likely impurities. Impurities more volatile than F_2 were removed by brief vacuum pumping on the liquid fluorine prior to transfer and at three intervals during transfer when the back pressure became high enough to stop the transfer. This treatment probably did not change the corrosive nature of fluorine.

(vi) When all the fluorine was transferred from the graduated tube to the capsule, the capsule fill port was sealed with a hydrogen-oxygen torch. Then the capsule was disconnected from the manifold, remove from the liquid helium and placed in liquid nitrogen for storage.

Because the graduated tube was maintained at 77 K throughout the transfer process, little or no CF_4, SiF_4, CO_2, or HF, whose vapor pressures are below 1 torr at 77 K, were transferred into the capsule. This purification might reduce the corrosiveness of the fluorine.

When charging the capsules, the fluorine control samples were charged first (14-16 June, 1976) and then the specimen capsules were charged in numerical order (17-25 June, 1976). On the average, three capsules were charged per day. It was observed that the first three fluorine control samples were darker in color than the remaining fluorine samples. After specimen 7525 was charged, the capsule loading was stopped for a day while the GF_2 was still pressurized in the manifold up to Valves 2 and 3. Upon resumption, after about 40 hours, when fluorine was condensed in the capsule containing specimen 7526, darker colored fluorine was again observed. Because of these observations the test plan was changed to add

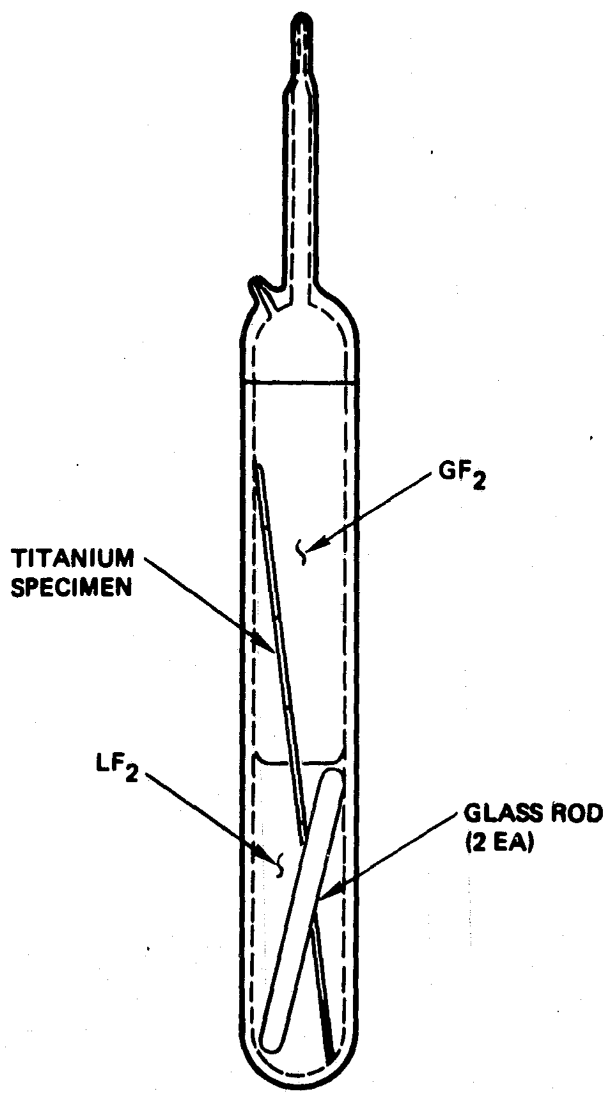

Figure 6. Loaded and Sealed Immersion Compatibility Capsule

post-immersion electron microprobe analysis of the residues after evaporation of fluorine from one capsule containing dark fluorine.

The glass capsules for LF_2 control specimens No. 7 and 8 would not seal properly, and were discarded without testing. The capsules for titanium specimens 7508 and 7511 failed by breaking at the side-arm during the initiation/loading process and the specimens were discarded. (7511 was suppose to be a replacement for 7508.) Specimen 7529 was a spare; it was sealed into a capsule but was not subjected to any additional operations. The specimens which were exposed to LF_2 prior to the capsule failing (7508 and 7511) become very corroded during evaporation of F_2 open to the humid atmosphere, and are unsuitable for further use. (See lower sketch on page F-3.)

There were a total of thirty fluorine-loaded capsules: eight capsules with 10 ml of liquid fluorine only and 22 capsules with a titanium specimen and 10 ml of liquid fluorine.

(b) Storage of Specimens and Liquid Fluorine

(i) A Cryo-flask was filled with LN_2. The storage cannisters were removed from the Cryoflask and placed in LN_2-filled Dewar flasks. The capsules for storage were placed in the cannisters, and when the cannisters were filled, they were replaced in the Cryoflask. A chart was prepared of the placement of the capsules; this chart is presented as Table X. The assignment of specimens to Group A and Group B is also given in Table X.

(ii) The loaded Cryoflask was transferred from the laboratory to an armored storage bunker with a supply of LN_2 refrigerant. LN_2 was replenished twice weekly, and the storage assembly was inspected regularly. The approved operating procedure, number D01755, for maintaining the test in storage, is presented as Appendix C of this report.

(c) Removal of Specimens and Fluorine Controls from Storage

The removal of the capsules from storage, the removal of fluorine from the capsules, and the analysis of the residue remaining after distillation of fluorine at 77 K was performed by following the approved operating procedure No. YS-28T-15, attached to this report as Appendix D.

(i) The capsules were removed from storage in the Cryo-flask and while still cooled by liquid nitrogen were attached to a ball valve by means of a Swagelok fitting and Teflon ferrules. The ball valve was in turn attached to the vacuum manifold shown in Figure 4. The ball valve had been drilled to permit the insertion of the capsule break-seal tip. The configuration is

TABLE X. SPECIMEN STORAGE LOCATION CHART

Specimen No.	Surface Finish	Heat Treated	Annealed	Initial Weight, g	Storage Canister No.	Test Group
7504	16	Yes	No	5.1380	3	B
7505	16	Yes	No	5.1570	3	B
7506	16	Yes	No	5.1780	3	B
7507	16	Yes	No	5.1023	3	B
7509	16	Yes	No	5.1368	4	A
7510	16	Yes	No	5.2066	4	A
7511	16	Yes	No	5.3021	4	A
7522	64	Yes	No	5.0853	4	A
7523	64	Yes	No	5.1962	5	B
7524	64	Yes	No	5.2110	5	B
7525	64	Yes	No	5.2240	5	B
7526	64	Yes	No	5.2286	5	B
7527	64	Yes	No	5.1724	6	A
7528	64	Yes	No	5.2931	6	A
7554	16	No	Yes	5.0616	7	A
7555	16	No	Yes	5.0880	7	A
7556	16	No	Yes	4.9442	7	B
7557	16	No	Yes	5.1087	7	B
7572	64	No	Yes	5.1401	8	A
7573	64	No	Yes	4.9885	8	A
7574	64	No	Yes	5.1323	8	B
7575	64	No	Yes	5.2082	8	B

Note: Canisters No. 1 and 2 contain four Group A and two Group B liquid fluorine reference samples, respectively.

shown in Figure 1 of Appendix D. The manifold and ball valve with the still-refrigerated capsule attached were evacuated for 15 minutes to remove any moisture adhering to the tip of the cold capsule. Then, with the vacuum pump isolated from the manifold, the ball valve was rotated to break the tip off the capsule. The pressure in the manifold rose to 320-330 torr. Again vacuum was applied to the manifold, but this time the gases were pumped through soda lime and charcoal scrubbers to avoid passing fluorine directly into the vacuum pump.

(ii) When all the fluorine had distilled from the capsule and been pumped from the manifold, an infrared gas cell with silver chloride windows was attached to the vacuum manifold and evacuated. The LN_2 Dewar was removed from the capsule, which then warmed to room temperature (298 K). The residue present at 77 K melted and became a gas and a sample was transferred into the evacuated gas cell. By means of infrared spectrometry, the composition of room temperature-volatile, 77 K involatile, evaporated residue was determined.

At room temperature while at reduced total pressure (1 to 10 torr) no involatile residue was seen in the capsules except for small quantities on the walls in capsules which had contained dark-colored fluorine.

(iii) After removal of all volatile materials, the capsule at 298 K was evacuated, back-filled to 1 atmosphere with helium and sealed for storage until the titanium specimen was examined.

The twenty-nine month (125 week) immersions were terminated over the period 13 to 17 November, 1978, and the thirty-nine month (168 week) immersions over the period 10 to 14 September 1979.

2.3.4.2 Characterization of Titanium

The analyses consisted of visual examination, both unaided and by microscope, scanning electron microscope (SEM) examination, weight change, metallographic examination to determine stress corrosion cracking and number/size of pits, and mechanical property tests (i.e., ultimate tensile strength, yield strength, and % elongation). The same test method and equipment were utilized throughout the test program.

Specimens were transported from the propellant laboratory to the metallurgical laboratory sealed in helium gas in the original capsules from the fluorine tests. Figures G-1 through G-6 in Appendix G are photographs of the still-sealed 39-month immersion capsules with the specimens still inside, in the state they were received at the metallurgical laboratory. The specimens were removed from the glass capsules,

weighed and placed in argon-filled specimen bottles. Samples of the brown residue adherent to the upper wall of the capsule containing specimen 7526 (see Figure 7) were removed for electron probe microanalysis. This capsule was one which had dark colored fluorine after the fluorine had been in contact with stainless steel tubing for about 40 hours.

When weight change data were compared, it was found that the mean gain for 39 months was less than for 29 months. It was postulated that perhaps the corrosion film had broken away from the coupon surfaces in the 39 month tests. The interiors of the empty capsules were then inspected carefully. Small amounts of loose, solid, flakey residues were observed at the bottoms of the 39 month capsules. A sample of residue was taken from capsule 7526 for electron microprobe analysis.

Tensile, SEM, pitting analysis, and metallographic test coupons were cut from the double dogbone specimens exposed to the liquid/vapor fluorine as shown in Figure 8. Room temperature (298 K) tensile tests were conducted with serrated V-grips which permitted the reduced area test section to be tested without any alterations. Tensile specimens tests at 77 K did require machining of the coupons prior to testing. The higher strengths at 77 K cause the coupons to be pulled from the serrated grips. In order to test the specimens at 77 K, the coupons were machined to the unsymmetrical configuration shown in Figure 9.

Photographs of the fractured coupons were taken after completion of the tensile tests.

SEM and metallographic/pitting analysis specimens were removed from the ends of the specimens because the liquid/vapor transition cannot be precisely determined in the middle of the coupon. Metallographic and pitting analysis specimens were plated with electroless nickel in aqueous solution to retain the specimen edges, because edges can become rounded during the cross-section preparation, making it difficult to focus optical instruments accurately on the edges. Photomicrographs were taken at 400 X and at 1000 X to show typical surface profiles of each specimen. This nickel coating is only weakly adherent to the titanium. In many cases it spalled off during sectioning; however it did protect the edges before it was lost. The figure on page G-27 shows an adherent coating; those on G-22 show a weakly adherent coating; in F-16 only remnants of the coating remain; and in F-17 it is completely gone. Quantitative pitting analysis was made by counting the number of pits and measuring the pit diameter (d) (width at the top of the pit) and pit depth (1) as well as pit

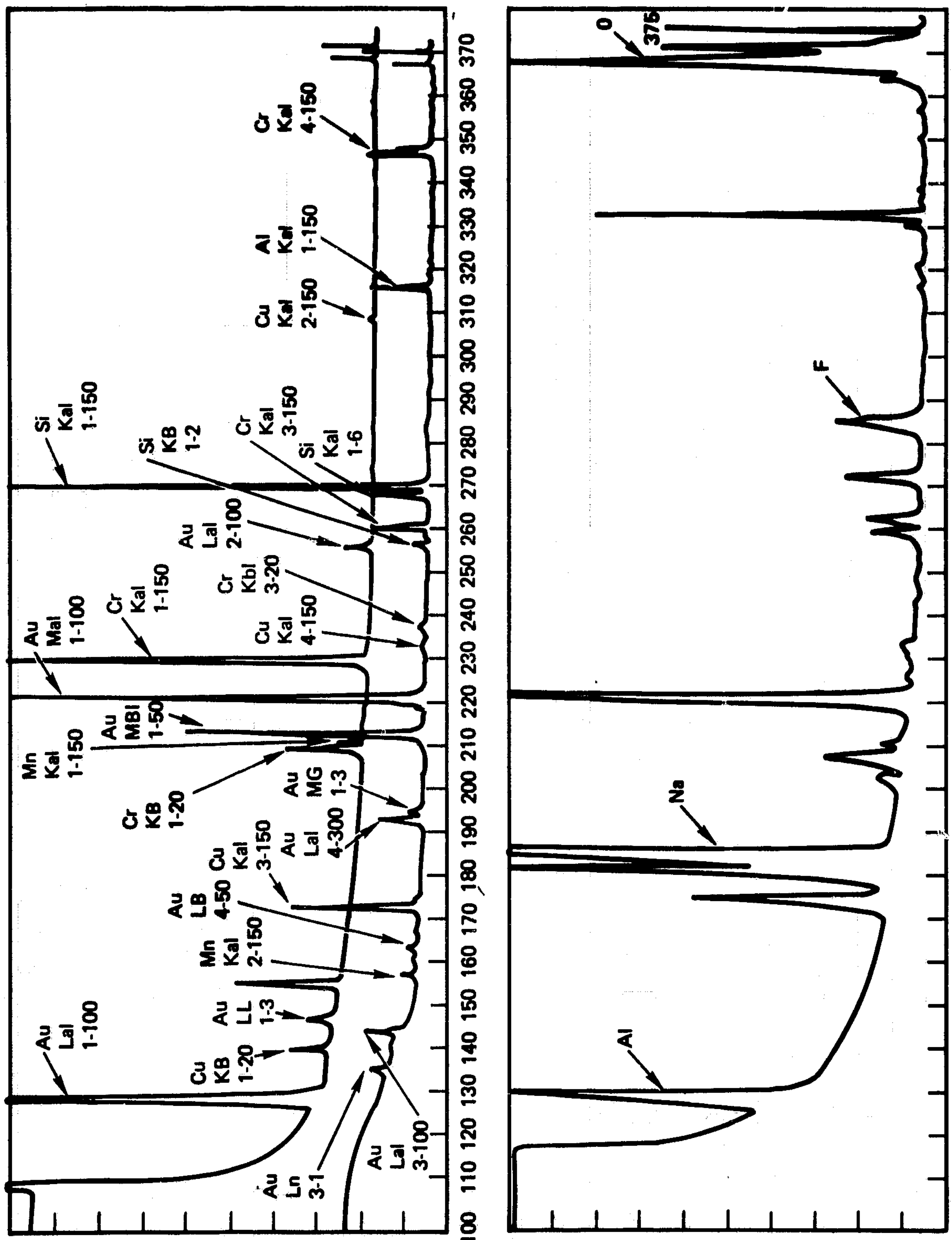

Figure 7. Electron Microprobe Traces of Dark Residue

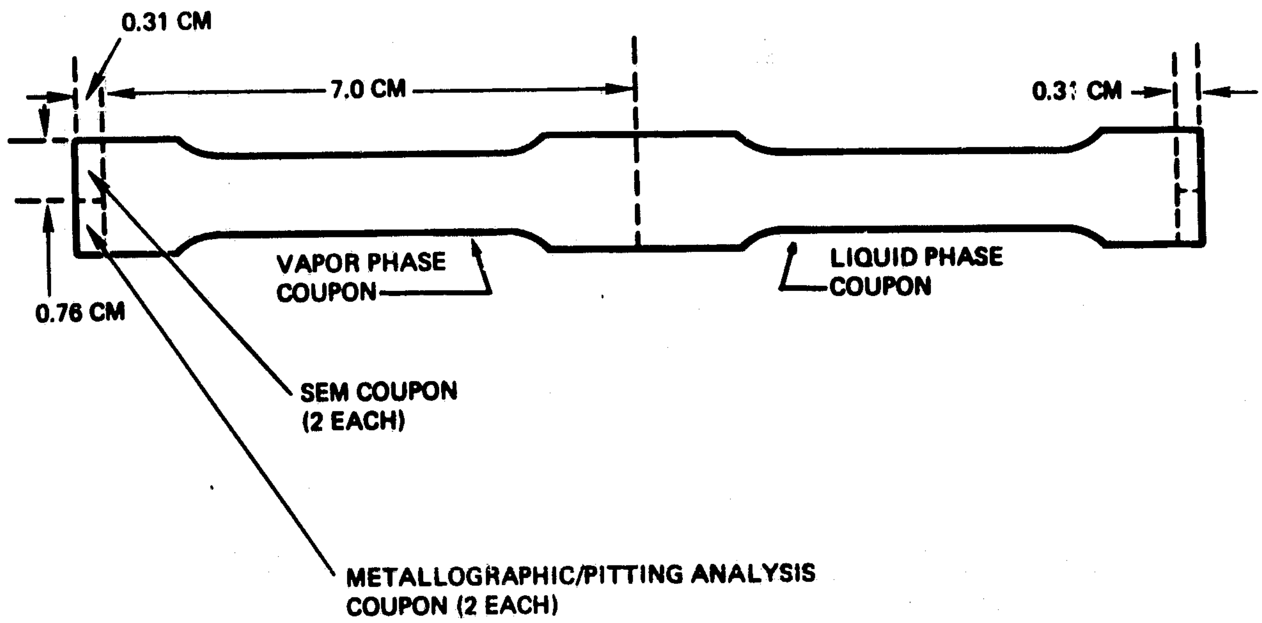

Figure 8. Test Coupon Locations on Immersion Specimen (See Figure 1 for complete dimensions)

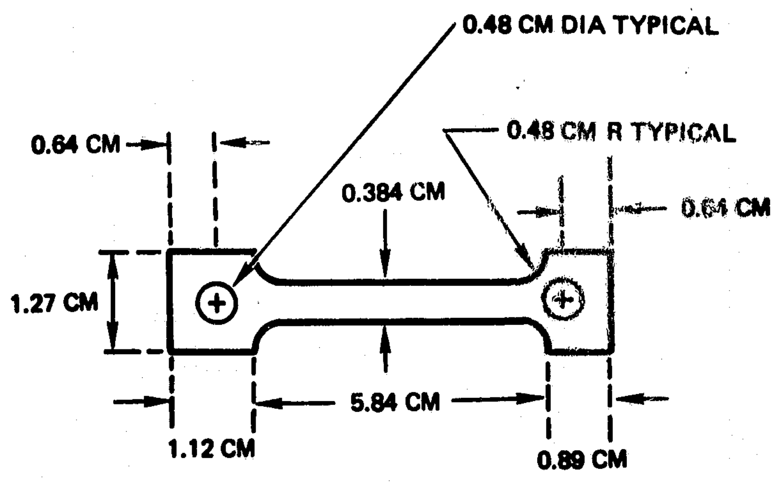

Figure 9. Modified Coupon for Cryogenic Tensile Test

spacing (distance between two adjacent pits). Data were obtained by random sampling and analysis of 0.254 cm of the linear distance of the cross-section, representing about one-sixth of the total width. Pit depth was measured with a metallograph.

The pit size was computed by assuming a circular cross-section and using the measured pit diameter data. By assuming uniform distribution, the pit concentration was calculated. Frequency distributions of pit depth, pit size, and pit depth/diameter ratio were calculated.

2.3.4.3 Program Performed

The pre-immersion characterization plan (Table 1) was modified as follows:

a) Mechanical property tests were conducted on 12 coupons prepared from 8 specimens. One coupon was tested from each of the unpassivated specimens (7500, 7520, 7550, and 7570), which was an addition to the plan. Eight F_2- passivated coupons, four each from 16 RMS heat-treated (7502 and 7503) and from 16 RMS annealed (7552 and 7553), were tested at room temperature (7502 and 7552) and two at LN_2 temperature (7503 and 7553).

b) Visual examinations, SEM, metallography and pitting analyses were carried out on all 12 pre-immersion specimens, instead of the 8 planned.

The 2-year duration post-immersion characterization plan (Table IV) was carried out, with the noted changes as follows:

a) The duration of the immersion test was increased to 29 months.

b) Mechanical property tests were conducted on 20 coupons prepared from all 10 specimens in the immersion test instead of the 16 coupons planned. Eight coupons were tested at 77 K, 4 from vapor immersion and 4 from liquid, and 12 at 298 K, 6 each from vapor and from liquid immersion. The first two of the cryogenic tests were failures, 7509 vapor and 7555 vapor, because the coupons had been machined to the wrong dimensions, and the fracture occurred in the grip region. The extra cryogenic tensile tests were added after this occurred.

c) All ten specimens were weighed, visually examined, and mapped, as planned.

d) Eight coupons were examined by SEM, as planned.

e) Sixteen coupons were examined metallographically, as planned.

f) Pitting analysis was carried out on eleven coupons, instead of the eight planned.

g) Only 2 control samples of LF_2 were stored for 24 months. (Compare Table III). See last paragraph of Section 2.3.4.1(a).

h) Chemical analyses of fluorine residues volatile at 298 K were carried out on 4 immersion samples and 2 controls, as planned.

The 3-year duration post-immersion characterization plan (Table V) was carried out as follows:

a) The duration of the immersion was increased to 39 months.

b) Mechanical property tests were conducted on 22 coupons prepared from all 11 specimens in the immersion test. Four coupons were tested at 77 K, 2 each from vapor and from liquid immersion. The remaining coupons were tested at 298 K.

c) All eleven specimens were weighed, visually examined, and mapped, as planned.

d) Eight coupons were examined by SEM, as planned.

e) Sixteen coupons were examined metallographically, as planned

f) Pitting analyses were carried out on eight coupons, as planned

g) Chemical analyses of 298 K-volatile residues from fluorine were carried out on eleven immersion samples and 4 controls, instead of 4 samples and 4 controls as planned.

h) Non-volatile residues in one capsule (specimen no. 7526) from dark fluorine were analyzed by electron microprobe.

2.3.5 Program Results - Data and Observations

2.3.5.1 Pre-Immersion Tests

a) Initial Fluorine Analysis: The data are reported in Table VII, Section 2.3.3.2.

b) Visual Examination, Mapping, Microscopic Examination and Scanning Electron Microscopy: The sketches and photographs from these activities are included in Appendix E, Figures E-1, E-4 through E-7.

All unpassivated specimens (7500, 7520, 7550, and 7570) are clean with no visible discolorations. All F_2-passivated specimens had slight discolorations. There was a contrast in surface morphology between 16 RMS and 64 RMS finishes, but there was no visible difference between heat-treated and annealed conditions. Under the microscope, there was definite surface etching and film formation on the passivated specimens.

c) Metallography and Pitting Analyses: Photomicrographs of the coupons are included in Appendix E, Figures E-2 and E-3.

The measurements of the locations and diameters of all pits encountered in the 0.254 cm length on each coupon are presented in Table XI. Distribution frequencies for pit depth, pit area, and depth/diameter ratio can be found in Tables E-I to E-III in Appendix E.

TABLE XI. PRE-IMMERSION PIT SIZES AND LOCATIONS

Coupon No.	Pit Nos.	Center-Center Pit Separation (cm $\times 10^{-4}$)	Diameter of Two Adjacent Pits (cm $\times 10^{-4}$)	Average Diameter of Two Adjacent Pits (cm $\times 10^{-4}$)
7500	1-2	64.90	2.03-0.76	1.40
	2-3	55.63	0.76-3.81	2.29
	3-4			
	4-5	166.75	2.54-0.76	1.65
	5-6	217.55	0.76-2.54	1.65
	6-7	99.06	2.54-2.54	2.54
	7-8	90.68	2.54-1.02	1.78
	8-9	36.58	1.02-1.02	1.02
7501	1-2	21.34	0.76-6.35	3.56
	2-3	106.55	6.35-1.02	3.69
	3-4	11.30	1.02-1.27	1.15
	4-5	2.92	1.27-2.54	1.91
	5-6	179.07	2.54-5.08	3.81
	6-7	63.88	5.08-0.76	2.92
	7-8	18.80	0.76-1.27	1.02
	8-9	121.29	1.27-2.54	1.91
	9-10	89.0	2.54-3.81	3.18
	10-11	7.49	3.81-1.02	2.42
	11-12	95.76	1.02-5.08	3.05
	12-13	18.29	5.08-1.02	3.05
7502	1-2	67.31	2.54-2.54	2.54
	2-3	248.84	2.54-2.54	2.54
	3-4	517.53	2.54-6.35	4.45
	4-5	29.85	6.35-2.54	4.45
	5-6	63.5	2.54-7.62	5.08
	6-7	407.67	7.62-2.54	5.08
	7-8	313.69	2.54-5.08	3.81
	8-9	8.26	5.08-1.27	3.18

(continued)

TABLE XI. PRE-IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No.	Pit Nos.	Center-Center Pit Separation (cm x10^{-4})	Diameter of Two Adjacent Pits (cm x10^{-4})	Average Diameter of Two Adjacent Pits (cm x10^{-4})
7502 (Cont.)	9-10	97.79	1.27-1.27	1.27
	10-11	(data lost)	1.27-2.54	1.91
	11-12	128.91	2.54-1.27	1.91
	12-13	106.68	1.27-1.27	1.27
	13-14	294.64	1.27-3.81	2.54
7503	1-2	8.30	2.54-3.81	3.18
	2-3	67.95	3.81-5.08	4.45
	3-4	48.26	5.08-5.08	5.08
	4-5	45.72	5.08-7.62	6.35
	5-6	171.45	7.62-5.08	6.35
	6-7	51.74	5.08-1.91	3.50
	7-8	42.85	1.91-2.54	2.23
	8-9	48.90	2.54-1.27	1.91
	9-10	80.65	1.27-12.7	6.99
	10-11	115.57	12.7-5.08	8.89
	11-12	74.93	5.08-10.16	7.62
	12-13	262.89	10.16-7.62	8.89
	13-14	514.35	7.62-5.08	6.35
	14-15	217.17	5.08-2.54	3.81
	15-16	142.24	2.54-2.54	2.54
7520	1-2	195.58	12.7-2.54	7.62
	2-3	71.63	2.54-1.02	1.78
	3-4	125.48	1.02-1.02	1.02
	4-5	131.32	1.02-2.54	1.78
	5-6	23.50	2.54-11.43	6.99
	6-7	12.7	11.43-1.27	5.72
	7-8	11.18	1.27-0.76	1.02
	8-9	33.40	0.76-5.08	2.92
	9-10	17.78	5.08-10.16	7.62

(continued)

TABLE XI. PRE-IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No.	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x10^{-4})
7520 (Cont.)	10-11	50.8	10.16-5.08	7.62
	11-12	6.99	5.08-3.81	4.45
	12-13	13.21	3.81-2.54	3.18
	13-14	132.08	2.54-2.54	2.54
	14-15	86.11	2.54-2.03	2.29
	15-16	156.59	2.03-3.81	2.92
	16-17	384.81	3.81-3.81	3.81
	17-18	337.19	3.81-1.27	2.54
7521	1-2	0		
	2-3	154.31	2.54-1.27	1.91
	3-4	90.55	1.27-2.03	1.65
	4-5	11.56	2.03-0.76	1.40
	5-6	37.85	6.76-1.27	1.02
	6-7	62.36	1.27-1.52	1.46
	7-8	100.46	1.52-1.27	1.40
	8-9	83.69	1.27-1.02	1.15
	9-10	88.52	1.02-0.76	0.89
	10-11	126.11	0.76-2.54	1.66
7550	1-2	551.18	6.35-3.81	5.08
	2-3	603.25	3.81-3.81	3.81
	3-4	142.24	3.81-1.27	2.54
	4-5	314.96	7.62-2.54	5.08
	5-6	66.04	2.54-2.54	2.54
	6-7	586.74	2.54-2.54	2.54
	7-8	196.22	2.54-8.89	5.72
	8-9	1098.55	8.89-3.81	6.35
7551	1-2	248.29	1.27-2.54	1.91
	2-3	266.70	2.54-2.54	2.54
	3-4	730.89	2.54-1.27	1.91

(continued)

TABLE XI. PRE-IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No.	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7552	1-2	226.82	1.52-7.62	4.57
	2-3	37.47	7.62-1.78	4.70
	3-4	33.27	1.78-1.27	1.53
	4-5	163.83	1.27-1.27	1.27
	5-6	79.88	3.81-1.02	2.42
	6-7	221.74	1.02-0.51	0.77
	7-8	19.56	0.51-0.51	0.51
	8-9	242.19	0.51-3.81	2.16
	9-10	26.04	3.81-2.54	3.18
	10-11	55.37	2.54-1.52	2.03
	11-12	23.62	1.52-2.54	2.03
7553	1-2	452.63	1.78-1.78	1.78
	2-3	11.56	1.78-1.02	1.40
	3-4	44.96	1.02-2.54	1.78
	4-5	106.43	2.54-2.03	2.29
	5-6	55.12	1.78-1.78	1.78
	6-7	295.53	1.78-2.54	2.16
	7-8	72.14	2.54-2.03	2.29
	8-9	402.34	2.03-2.54	2.29
7570	1-2	128.91	3.81-10.16	6.99
	2-3	74.93	10.16-2.54	6.35
	3-4	123.83	2.54-6.35	4.45
	4-5	288.04	6.35-0.76	3.56
	5-6	140.72	0.76-1.27	1.02
	6-7	574.68	3.81-2.54	3.18
	7-8	484.51	2.54-1.27	1.91

(continued)

TABLE XI. PRE-IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No.	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7571	1-2	196.22	5.08-1.27	3.18
	2-3	303.15	1.27-2.54	1.98
	3-4	383.54	2.54-2.54	2.54
	4-5	40.01	2.54-1.27	1.91
	5-6	195.83	1.27-1.27	1.27
	6-7	454.28	2.54-1.78	2.16
	7-8	77.98	1.78-1.29	1.53

TABLE XII. SUMMARY OF DIMENSIONS AND FREQUENCIES OF PITS IN PRE-IMMERSION SPECIMENS

Specimen Number	Number of Pits Counted in 0.254 cm	Pit Frequency No. $x10^3/cm^2$	Pit Depth cm $x10^{-4}$ Mean	Pit Depth cm $x10^{-4}$ Standard Dev.	Pit Area cm^2 $x10^{-7}$ Mean	Pit Area cm^2 $x10^{-7}$ Standard Dev.	Depth/Diameter Ratio Mean	Depth/Diameter Ratio Standard Dev.
Unpassivated								
7500	9	1.3	0.89	0.21	0.36	0.13	0.68	0.26
7520	18	5.0	2.00	0.95	2.32	3.45	0.69	0.40
7550	10	1.6	1.54	0.88	1.9	2.0	0.62	0.15
7570	9	1.3	2.03	0.83	1.7	2.6	0.83	0.23
Passivated								
7501	13	2.6	1.21	0.74	0.77	1.02	0.76	0.27
7502	14	3.0	0.78	0.32	1.03	1.33	0.34	0.14
7503	16	4.0	1.32	0.69	2.73	3.38	0.40	0.19
7521	11	1.9	1.26	0.67	0.20	0.17	1.10	0.69
7551	5	0.4	1.48	0.70	0.48	0.42	0.83	0.23
7552	13	2.6	1.06	0.57	0.68	1.2	0.66	0.46
7553	10	1.6	1.09	0.40	0.33	0.14	0.70	0.18
7571	9	1.3	1.46	0.77	0.48	0.61	0.82	0.19
Simple Mean	11.4	2.2	-	-	-	-	-	-
Weighted Mean	-	-	1.34	-	1.23	-	0.67	-
Range	5-18	0.4 - 5.0	0.78-2.03		0.20-2.73		0.40-1.10	

A summary of the average surface characteristics for each coupon is given in Table XII. It includes mean values, calculated from the original data, for number of pits per 0.254 cm length, number of pits per unit 1 cm^2 area, mean pit depth, mean pit area, and mean depth/diameter ratio. In addition, the Table contains overall averages for the above parameters for all coupons in the pre-immersion test. These are weighted "means of means" which were calculated by multiplying the mean value for each coupon in the table by the number of pits per unit length, summing the products, and dividing the product sum by the total number of pits for all coupons.

d) Mechanical Properties (Tensile Tests): The data obtained in the tensile tests are presented in Table XIII. Photographs of the fractured coupons are in Appendix E, Figure E-8 through E-10.

2.3.5.2 Immersion Storage

The storage of the capsules under LN_2 over the complete program took place without incident. All capsules placed in test were recovered intact at the ends of the immersion periods. Actual immersion durations were 29 months for Group A and 39 months for Group B.

TABLE XIII. TENSILE MECHANICAL PROPERTIES OF PRE-IMMERSION TITANIUM COUPONS

Coupon No.	Test Temp (K)	Ultimate Strength (psi)	Yield Strength 0.2% Offset (psi)	% Elongation 1/2 Inch
7500-1	298	176,600	165,200	10
7520-1	298	173,800	159,400	13
7550-1	298	147,200	138,200	16
7570-1	298	146,500	139,100	16
7502-1	298	175,800	161,900	10
7502-2	298	175,000	163,300	12
7503-1	77	256,400	-	6
7503-2	77	256,000	-	6
7552-1	298	146,700	138,900	16
7552-2	298	147,300	139,600	16
7553-1	77	231,700	-	9
7553-2	77	225,800	-	8

-1 coupon is from end of specimen with Vee notch.

-2 coupon is from plain end of specimen.

2.3.5.3 Post 29-Month Immersion Tests

a. Fluorine Impurity Analysis: The results from these analyses for volatile impurities are presented in Table XIV. No nonvolatile residues were observed in any of the capsules.

b. Visual Examinations, Mapping, Microscopic Examination, and Scanning Electron Microscopy: The sketches and photographs from these activities are included in Appendix F, Figures F-1, F-2 and F-4.

All surfaces had irregular surface films, which usually were thicker on the liquid-immersed portion. However, sharp demarcations between the parts immersed in liquid and in vapor were not detected. The films appeared to be more continuous on the annealed specimens than on the heat-treated ones for both surface finishes. Discrete corrosion-product particles were more prevalent on the heat-treated specimens.

c. Weight/Weight Changes: The weight change data are presented in Table XV.

The test specimens (including both the top and bottom of the double dogbone specimens) had an average weight gain of 0.0181 g (range of 0.0139 to 0.0223 g) with an average percent weight gain of 0.35% (range of 0.27 % to 0.50 %). When the percent weight change data are examined, it appears that specimen 7527 is out of line with the remainder. As a test, the arithmetic mean and standard deviation for all specimens were calculated: $\overline{X} = 0.352$; $\sigma_n = 0.068$. Since the value for the percent change for specimen 7527, 0.50, is greater than $\overline{X} + 2\sigma = 0.487$, the probability is greater than 95% that the datum for 7527 represents a different population, so it is ignored in the analyses.

d. Metallography and Pitting Analysis: Photomicrographs of the coupons are included in Appendix F, Figure F-3.

The measurements of the locations and diameters of all pits encountered in the 0.254 cm length on each coupon are presented in Table XVI. Distribution frequencies for pit depth, pit area, and depth/diameter ratio can be found in Tables F-I to F-III in Appendix F.

A summary of the average surface characteristics for each coupon is given in Table XVII. It includes mean values, calculated from the original data, for number of pits per 0.254 cm length, number of pits per unit 1 cm^2 area, mean pit depth, mean pit area, and mean depth/diameter ratio.

TABLE XIV. ANALYSIS OF VOLATILE RESIDUES
29 MONTH IMMERSION

Specimen No,	Residue Content							
	CO_2		CF_4		HF		SiF_4	
	mg	% v/v	mg	% v/v	mg	% v/v	mg	% v/v
F_2 Control #5	<0.01	$<5.6 \times 10^{-5}$	<0.01	$<2.8 \times 10^{-5}$	<0.04	$<1.1 \times 10^{-2}$	<0.01	$<4.9 \times 10^{-4}$
F_2 Control #6	<0.01	$<5.6 \times 10^{-5}$	<0.01	$<2.8 \times 10^{-5}$	<0.04	$<1.1 \times 10^{-2}$	<0.01	$<4.9 \times 10^{-4}$
7505	0.05	2.8×10^{-4}	<0.01	$<2.8 \times 10^{-5}$	<0.04	$<1.1 \times 10^{-2}$	<0.01	$<4.9 \times 10^{-4}$
7522	0.41	2.3×10^{-3}	<0.01	$<2.8 \times 10^{-5}$	<0.04	$<1.1 \times 10^{-2}$	0.10	4.9×10^{-3}
7555	0.35	2.0×10^{-3}	<0.01	$<2.8 \times 10^{-5}$	<0.04	$<1.1 \times 10^{-2}$	<0.01	$<4.9 \times 10^{-4}$
7575	0.41	2.3×10^{-3}	<0.01	$<2.8 \times 10^{-5}$	<0.04	$<1.1 \times 10^{-2}$	<0.01	$<4.9 \times 10^{-4}$
Lower Limit of Detection	0.01	5.6×10^{-5}	0.01	2.8×10^{-5}	0.04	1.1×10^{-2}	0.01	4.9×10^{-4}

Notes: Capsules initially loaded with 15.5 g of LF_2.
Mg indicates mg of material collected in one capsule.
% v/v indicates volume % of material in total F_2 in gas phase

TABLE XV. POST IMMERSION WEIGHT CHANGES
29-MONTH IMMERSION

Specimen I.D.	Specimen Cond.	Pre-Immersion Weight (g)	Post-Immersion Weight (g)	Weight* Gain Δg	Weight* Gain %	$\overline{X}$	σ_n	Mean % Gains $\overline{X}$	Mean % Gains σ_n
7505	HT 16	5.1570	5.1718	+0.0148	0.29				
7509	HT 16	5.1368	5.1530	+0.0162	0.32	0.307	0.0203		
7510	HT 16	5.2066	5.2205	+0.0139	0.27				
7522	HT 64	5.0853	5.1018	+0.0165	0.32				
7527	HT 64	5.1724	5.1982	+0.0258	0.50**	0.313	0.0111	0.313	0.0266
7528	HT 64	5.2931	5.3091	+0.0160	0.30				
7555	A 16	5.0880	5.1058	+0.0178	0.35	0.343	0.0066		
7557	A 16	5.1087	5.1259	+0.0172	0.34				
7572	A 64	5.1401	5.1608	+0.0207	0.40	0.415	0.0127		
7575	A 64	5.2082	5.2305	+0.0223	0.43				

*Weights are for complete specimens, including both vapor-immersed and liquid-immersed portions.

**Outlier: not included in calculations

TABLE XVI. 29-MONTH IMMERSION PIT SIZES AND LOCATIONS

Coupon No. and Phase	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7505 LIQUID	1- 2	69.9	6.4-14.5	10.5
	2- 3	139.1	14.5- 6.6	10.6
	3- 4	30.3	6.6-11.4	9.0
	4- 5	15.4	11.4- 8.1	9.8
	5- 6	15.2	8.1- 2.5	5.3
	6- 7	24.7	2.5- 5.6	4.1
	7- 8	23.5	5.6- 5.8	5.7
	8- 9	38.9	5.8- 5.3	5.4
	9-10	9.7	5.3- 9.1	7.2
	10-11	21.1	9.1-10.2	9.7
	11-12	14.9	10.2- 9.4	9.8
7505 VAPOR	1- 2	69.0	4.1- 5.3	4.7
	2- 3	37.1	5.3- 4.8	5.1
	3- 4	48.9	4.8- 1.5	3.2
	4- 5	88.1	1.5- 7.1	4.3
7510 LIQUID	1- 2	39.2	2.5- 7.6	16.3
	2- 3	34.3	7.6- 3.6	5.6
	3- 4	48.3	3.6- 5.1	4.4
	4- 5	68.6	5.1- 7.6	6.4
	5- 6	14.5	7.6- 8.1	7.9
	6- 7	11.9	8.1- 7.1	7.6
	7- 8	63.1	7.1- 5.8	6.5
	8- 9	22.6	5.8- 6.4	6.1
	9-10	42.7	6.4- 9.4	7.9
	10-11	87.2	9.4- 6.4	7.9
7510 VAPOR	1- 2	31.0	6.9- 7.9	7.4
	2- 3	127.6	7.9-14.0	11.0
	3- 4	24.4	14.0- 6.4	10.2
	4- 5	8.9	6.4- 6.4	6.4

(continued)

TABLE XVI. 29-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7510	5- 6	50.8	6.4-10.4	8.4
VAPOR (Cont'd)	6- 7	150.4	10.4- 3.3	6.9
	7- 8	42.0	3.3- 8.4	5.9
	8- 9	87.8	8.4- 9.7	9.1
7527	1- 2	35.9	17.8-15.8	16.8
LIQUID	2- 3	18.[illegible]	15.8- 7.6	11.7
	3- 4	33.3	7.6-12.7	10.2
	4- 5	41.6	12.7-23.6	18.2
	5- 6	46.4	23.6-29.0	26.3
	6- 7	68.6	29.0-25.4	27.2
	7- 8	254.0	25.4-25.4	25.4
	8- 9	137.2	25.4-25.4	25.4
	9-10	97.9	25.4-18.0	21.7
	10-11	80.8	18.0-19.1	18.6
7527	1- 2	26.2	21.6-21.1	21.4
VAPOR	2- 3	24.9	21.1-24.1	22.6
	3- 4	22.6	24.1-17.0	20.6
	4- 5	20.0	17.0-13.7	15.4
	5- 6	21.9	13.7-18.8	16.3
	6- 7	30.9	18.8-17.5	18.2
	7- 8	22.2	17.5-17.3	17.4
	8- 9	16.2	17.3- 6.4	11.9
	9-10	34.2	6.4-13.7	10.1
	10-11	81.7	13.7- 6.4	10.1
	11-12	135.7	6.4- 7.9	7.2
	12-13	54.7	7.9- 3.8	5.9
	13-14	7.7	3.8- 3.3	3.6
	14-15	15.7	3.3- 4.6	4.0
	15-16	136.3	4.6-71.9	38.3
	16-17	134.4	71.9- 8.9	40.4

(continued)

TABLE XVI. 29-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7557	1- 2	6.8	2.5- 6.1	4.3
LIQUID	2- 3	20.9	6.1-11.9	9.0
	3- 4	65.5	11.9-10.9	11.4
	4- 5	44.3	10.9- 5.1	8.0
	5- 6	27.1	5.1- 9.4	7.3
	6- 7	40.6	9.4-56.6	33.0
	7- 8	91.3	56.6- 7.6	32.1
	8- 9	11.0	7.6- 5.1	6.4
	9-10	19.8	5.1- 6.4	5.8
	10-11	43.6	6.4- 4.6	5.5
	11-12	56.7	4.6- 2.5	3.6
7557	1- 2	24.3	2.0- 4.3	3.2
VAPOR	2- 3	17.3	4.3- 1.8	3.1
	3- 4	26.0	1.8- 4.3	3.1
	4- 5	34.4	4.3- 2.5	3.4
	5- 6	6.4	2.5- 6.6	4.6
	6- 7	10.7	6.6- 2.0	4.3
7572	1- 2	26.2	21.6-21.1	21.4
VAPOR	2- 3	24.9	21.1-24.1	22.6
	3- 4	22.6	24.1-17.0	20.6
	4- 5	20.0	17.0-13.7	15.4
	5- 6	29.0	13.7-18.8	16.3
	6- 7	100.0	18.8-18.5	18.7
	7- 8	36.6	18.5-11.4	15.0
	8- 9	142.1	11.4-20.3	15.9
	9-10	35.5	20.3- 6.4	13.4
	10-11	21.5	6.4-15.2	10.8
	11-12	23.5	15.2-14.0	14.6

(continued)

TABLE XVI. 29-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7572 VAPOR (Cont'd)	12-13	236.3	14.0-14.0	14.0
	13-14	121.3	14.0-10.2	12.1
	14-15	61.7	10.2- 7.6	8.9
	15-16	259.7	7.6- 6.4	7.0
7575 LIQUID	1- 2	13.2	6.6-12.5	9.6
	2- 3	32.0	12.5-12.2	12.4
	3- 4	15.8	12.2- 4.6	8.4
	4- 5	98.7	4.6- 4.3	4.5
	5- 6	14.6	4.3- 8.1	6.2
	6- 7	175.1	8.1-14.5	11.3
	7- 8	214.7	14.5- 6.4	10.5
	8- 9	108.8	6.4- 6.4	6.4
	9-10	13.6	6.4- 5.6	6.0
	10-11	11.6	5.6- 3.8	4.7
	11-12	20.7	3.8- 3.6	3.7
	12-13	30.9	3.6-10.9	7.3
	13-14	16.8	10.9- 8.9	9.9
	14-15	45.2	8.9-10.2	9.6
	15-16	36.1	10.2- 3.6	6.9
	16-17	170.1	3.6- 7.9	5.8
	17-18	34.6	7.9- 2.8	5.4
	18-19	117.8	2.8- 8.1	5.5
7575 VAPOR	1- 2	15.9	12.7- 3.8	8.3
	2- 3	14.9	3.8-13.7	8.8
	3- 4	38.8	13.7-18.0	15.9
	4- 5	21.4	18.0-16.5	17.3
	5- 6	22.5	16.5- 5.1	10.8
	6- 7	39.3	5.1-27.2	16.2
	7- 8	75.6	27.2- 8.1	17.7

(continued)

TABLE XVI. 29-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pit Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7575	8- 9	48.0	8.1-25.4	16.8
VAPOR (Cont'd)	9-10	53.5	25.4-10.9	18.2
	10-11	25.4	10.9- 9.1	10.0
	11-12	92.6	9.1-25.4	17.3
	12-13	38.2	25.4- 4.8	15.1
	13-14	31.4	4.8- 7.9	6.4
	14-15	32.6	7.9- 4.1	6.0
	15-16	81.9	4.1- 5.6	4.9
	16-17	28.4	5.6- 5.1	5.4
	17-18	70.6	5.1-18.0	11.6
	18-19	52.2	18.0-18.5	18.3
	19-20	29.1	18.5- 7.1	12.8
	20-21	13.5	7.1-10.4	8.8
	21-22	94.9	10.4- 8.4	9.4
	22-23	10.4	8.4- 7.6	8.0
	23-24	84.1	7.6- 4.6	6.1
	24-25	19.1	4.6-12.7	8.7
	25-26	80.4	12.7-10.2	11.5
	26-27	21.8	10.2-17.3	13.8
	27-28	23.0	17.3- 2.5	9.9
	28-29	87.4	2.5- 6.9	4.7
	29-30	42.0	6.9- 8.9	7.9
	30-31	8.1	8.9- 3.8	6.4
	31-32	24.3	3.8- 5.8	4.8
	32-33	48.4	5.8- 5.8	5.8
	33-34	48.4	5.8- 9.7	7.8

TABLE XVII. SUMMARY OF DIMENSIONS AND FREQUENCIES OF PITS IN 29-MONTH IMMERSION COUPONS

Coupon Number	Number of Pits Counted in 0.254 cm	Pit Frequency No. $x10^3/cm^2$	Pit Depth cm $x10^{-4}$		Pit Area cm^2 $x10^{-7}$		Depth/Diameter Ratio	
			Mean	Standard Dev.	Mean	Standard Dev.	Mean	Standard Dev.
7505 Liquid	12	2.2	2.57	1.32	5.66	4.44	0.35	0.16
7505 Vapor	5	0.4	2.02	0.58	1.89	1.38	0.51	0.21
7510 Liquid	11	1.9	1.51	0.58	3.43	1.87	0.26	0.12
7510 Vapor	9	1.3	2.16	1.32	5.86	4.25	0.26	0.12
7527 Liquid	11	1.9	7.37	5.40	34.30	19.20	0.36	0.17
7527 Vapor	17	4.5	4.46	3.62	39.50	95.50	0.31	0.12
7557 Liquid	12	2.2	1.58	0.37	24.70	71.50	0.27	0.16
7557 Vapor	7	0.8	1.74	0.91	1.10	1.15	0.58	0.36
7572 Vapor	16	4.0	6.02	5.59	20.00	13.00	0.38	0.26
7575 Liquid	19	5.6	4.34	3.32	5.19	4.50	0.73	0.90
7575 Vapor	34	17.9	7.22	6.27	12.20	15.10	0.99	1.60
Simple Mean	13.9	3.9	-	-	-	-	-	-
Weighted Mean	-	-	4.50	-	15.4	-	0.54	-
Range	5-34	0.4 - 17.9	1.51-7.37		1.1-39.5		0.26-0.99	

In addition, the Table contains overall averages for the above parameters for all coupons in the pre-immersion test. These are weighted "means of means" which were calculated by multiplying the mean value for each coupon in the table by the number of pits per unit length, summing the products, and dividing the product sum by the total number of pits for all coupons.

The data on pit frequency, pit depth, and pit diameter do not indicate any consistent pattern of increased attack in either of vapor or liquid fluorine. However, the annealed coupons are more severely attacked than the heat treated coupons, and the 64 RMS finish more than the 16 RMS.

e. Mechanical Properties (Tensile Tests): The data obtained in the tensile tests are presented in Table XVIII. Photographs of the fractured coupons are in Appendix F, Figures F-5 and F-6.

The coupons which broke in the holding fixture at 77 K were the wrong configuration for the low temperature mechanical tests - - they had not been properly machined to the shape shown in Figure 9. An intermediate configuration, with insufficient reduction in area, was used - - see the top parts of the figures on pages F-31 and F-32. The failures were not related to the exposure to fluorine.

TABLE XVIII. TENSILE MECHANICAL PROPERTIES OF 29-MONTH IMMERSION TITANIUM COUPONS

Coupon No.	Test Temp (K)	Ultimate Strength (psi)	Yield Strength 0.2% Offset	% Elongation 1/2 Inch
7505-T	298	177,000	166,500	16
7505-B	298	173,100	163,500	14
7509-T*	77	-	-	-
7509-B	298	174,400	165,100	16
7510-T	298	177,800	167,900	16
7510-B	77	244,400	-	8
7522-T	298	174,600	164,300	14
7522-B	298	175,200	165,200	16
7527-T	77	251,000	-	5
7527-B	77	254,200	-	5
7528-T	298	177,500	166,800	14
7528-B	298	177,000	166,200	16
7555-T*	77	-	-	-
7555-B	298	146,200	139,100	20
7557-T	298	150,400	147,200	16
7557-B	77	221,500	-	12
7572-T	77	250,000	-	6
7572-B	77	224,000	-	7
7575-T	298	149,600	140,100	18
7575-B	298	149,200	141,300	18

NOTE: T=Top; Vapor Phase F_2 Immersion Exposure.
B=Bottom; Liquid Phase F_2 Immersion Exposure.

* Specimen broke in holding fixture.

2.3.5.4 Post 39-Month Immersion Tests

a. Fluorine Impurity Analysis: The results from these analyses for volatiles are presented in Tables XIX.

All residues from both controls and specimens have measurable quantities of CO_2; the mean for all 15 is 1.70 X 10^{-3} % v/v. Measurable quantities of SiF_4 were noted in all control samples (mean = 10.3 X 10^{-3} % v/v, range = 2.5 - 20 X 10^{-3}) but the range was surprisingly wide. However, only two of the titanium immersion capsules had appreciable SiF_4, 7523 and 7526. Contents of both CF_4 and HF were below the detectable limit in all capsules.

b. Analysis of Non-Volatile Residues: These analyses are reported in Table XX. The red-brown residue from the "dark" fluorine contains large quantities of chormium, and some copper, aluminum, and manganese. The only source of chromium in the system is the 18-8 CRES tubing in the manifold, and chromium fluorides are highly colored and are volatile at 298 K.

The flaky residue from the bottom of capsule 7526 contains significant quantities of titanium. It should be noted that the specimem in the capsule from which it was taken had the second highest weight gain of all 39-month specimens. Further analyses of these residues and attempts to determine weights of the loose material were not conducted because the contents had not been protected from access of the moist atmosphere after removal of the specimens.

c. Visual Examination, Mapping, Microscopic Examination, and Scanning Electron Microscopy: The sketches and photographs from these activities are included in Appendix G, Figures G-1 through G-3 and G-5 through G-20.

Visual examinations were performed at magnifications up to 40X on all 11 double dogbone specimens from the 39 months exposure, see Figure G-3. Small pits appear to be present in the surface of some of the specimens, see Figure G-4-3. Test coupons from two double dogbone specimens were examined on the JOEL Model JSM-2 scanning electron microscope. Both liquid and vapor exposed areas of the specimens were examined. The areas exposed to the liquid fluorine appeared to have a heavier scale than the areas exposed to the fluorine vapor (see Figure G-5 throug G-20). It should be noted that the vapor exposed area of specimen 7573 has very little corrosion products (Figure G-17 and G-18) whereas liquid exposed specimen 7523 (Figures G-11 and G-12) exhibited thick layer of deposits.

d. Weight/Weight Changes: The weight change data are incorporated in Table XXI.

These data are suspect and should not be used for mathematical analyses, because of the detection of titanium salt residues in the emptied capsules. No specific reason for flaking of the corrosion film can be cited. The possibility of severe vibration or shock during transit from the propellant laboratory to the metallurgical laboratory cannot be ruled out, as no requirement for special treatment during transport was imposed. On the other hand, spontaneous cracking and flaking of the corrosion film over a longer formation period and the more severe pitting attack in the titanium is also possible.

e. Metallography and Pitting Analysis: Photomicrographs of the coupons are included in Appendix G, Figures G-4 and G-23.

The measurements of the locations and diameters of all pits encountered in the 0.254 cm length on each coupon are presented in Table XXII. Distribution frequencies for pit depth, pit area, and depth/diameter ratio can be found in Tables G-I to G-III in Appendix G.

A summary of the average surface characteristics for each coupon is given in Table XXIII. It includes mean values, calculated from the original data, for number of pits per 0.254 cm length, number of pits per unit 1 cm^2 area; mean pit depth, mean pit area, and mean depth/diameter ratio. In addition, the Table contains overall averages for the above parameters for all coupons in the pre-immersion test. These are weighted "means of means" which were calculated by multiplying the mean value for each coupon in the table by the number of pits per unit length, summing the products, and diving the product sum by the total number of pits for all coupons.

The data show that pit frequency is not dependent on fluorine phase, or alloy heat treat condition. However, both pit depth and pit area are strong functions of initial surface finish, with the 64 RMS material yielding deeper and wider pits.

f. Mechanical Properties (Tensile Tests): The data obtained in the tensile tests are presented in Table XXIV. Photographs of the fractured coupons are in Appendix G, Figures G-21 and G-22.

No correlations of mechanical properties with immersion parameters were found.

TABLE XIX. ANALYSES OF VOLATILE RESIDUE IN 39-MONTH CAPSULES

Specimen No.	Residue Content							
	CO_2		CF_4		HF		SiF_4	
	Mg	% v/v	mg	% v/v	mg	% v/v	mg	% v/v
Fluorine Control 1	0.90	5.0×10^{-3}	<0.01	$<2.8 \times 10^{-5}$	<0.04	$<1.1 \times 10^{-2}$	0.05	2.5×10^{-3}
Fluorine Control 2	0.45	2.5×10^{-3}	<0.01		<0.04		0.40	2.0×10^{-2}
Fluorine Control 3	0.18	1.0×10^{-3}	<0.01		<0.04		0.20	9.8×10^{-3}
Fluorine Control 4	0.20	1.1×10^{-3}	<0.01		<0.04		0.18	8.8×10^{-3}
7504	0.20	1.1×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7506	0.20	1.1×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7507	0.25	1.4×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7523	0.40	2.2×10^{-3}	<0.01		<0.04		0.40	2.0×10^{-2}
7524	0.35	2.0×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7525	0.25	1.4×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7526	0.30	1.7×10^{-3}	<0.01		<0.04		0.35	1.1×10^{-3}
7554	0.30	1.7×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7556	0.15	8.4×10^{-4}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7573	0.20	1.1×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
7574	0.25	1.4×10^{-3}	<0.01		<0.04		<0.01	$<4.9 \times 10^{-4}$
Lower Limit of Detection	0.01	5.6×10^{-5}	0.01	2.8×10^{-5}	0.04	1.1×10^{-2}	0.01	4.9×10^{-4}

Notes: Capsules initially loaded with 15.5 g of LF_2.
Mg indicates mg of material collected in one capsule
% v/v indicates volume % of material in total F_2 in capsule totally in gas phase

TABLE XX. ANALYSES OF NON-VOLATILE RESIDUES IN CAPSULE 7526

ELECTRON PROBE MICROANALYSIS OF TUBE RESIDUES (SEMI-QUANTITATIVE)

RED-BROWN RESIDUE ON WALL OF TUBE

ELEMENT	SEMIQUANTITATIVE CONCENTRATION ESTIMATE
FLUORINE	5 to 10%
CHROMIUM	> 10%
COPPER	5 to 10%
ALUMINUM	1 to 5 %
MANGANESE	1 to 5 %

WHITE FLAKES AT BOTTOM OF TUBE

ELEMENT	SEMIQUANTITATIVE CONCENTRATION ESTIMATE
CHROMIUM	> 10%
COPPER	> 10%
ALUMINUM	1 to 5%
MANGANESE	1 to 5%
TITANIUM	1 to 5%
TIN	$\leq$ 1%
ZINC	1 to 5%

TABLE XXI. POST 39-MONTH IMMERSION WEIGHT CHANGE MEASUREMENTS

Specimen No.	Specimen Cond.	Pre-Immersion Weight (g)	Post-Immersion Weight (g)	Weight Change Δg	Weight Change %	$\overline{X}$	Mean % Change σ_n	Mean % Change $\overline{X}$	σ_n
7504	HT 16	5.1380	5.1409	+0.0029	0.06				
7506	HT 16	5.1780	5.1845	+0.0065	0.13	+0.087	0.038		
7507	HT 16	5.1023	5.1061	+0.0038	0.07				
7523	HT 64	5.1962	5.1976	+0.0014	0.03				
7524	HT 64	5.2110	5.2191	+0.0081	0.15	+0.163	0.099		
7525	HT 64	5.2240	5.2349	+0.0109	0.21			+0.134	0.099
7526	HT 64	5.2286	5.2423	+0.0137	0.26				
7554	A 16	5.0616	5.0790	+0.0174	0.34	+0.205	0.191		
7556	A 16	4.9442	4.9475	+0.0033	0.07				
7573	A 64	4.9885	4.9933	+0.0048	0.10	+0.075	0.035		
7574	A 64	5.1323	5.1347	+0.0024	0.05				

$\overline{X}$ = Mean

σ_n = Standard Deviation

TABLE XXII. 39-MONTH IMMERSION PIT SIZES AND LOCATIONS

Coupon No. and Phase	Pits Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7504	1- 2	8.5	3.6- 3.8	3.7
LIQUID	2- 3	5.7	3.8- 5.1	4.5
	3- 4	9.7	5.1-14.2	9.7
	4- 5	17.0	14.2- 7.6	10.9
	5- 6	20.3	7.6-22.9	15.3
	6- 7	26.3	22.9- 8.9	15.9
	7- 8	11.0	8.9- 3.1	6.0
	8- 9	41.7	3.1-20.3	11.7
	9-10	153.2	20.3-37.1	28.7
	10-11	73.8	37.1- 8.9	23.0
	11-12	25.3	8.9-11.2	10.1
	12-13	26.5	11.2- 6.4	8.8
	13-14	31.8	6.4-47.0	26.7
	14-15	35.3	47.0- 5.8	26.4
	15-16	10.3	5.8- 5.1	5.5
	16-17	53.3	5.1-19.8	12.5
	17-18	79.8	19.8-25.4	22.6
	18-19	19.6	25.4- 8.6	17.0
	19-20	11.7	8.6-14.7	11.7
	20-21	31.4	14.7-31.8	23.3
	21-22	79.4	31.8-25.4	28.6
	22-23	38.4	25.4- 9.7	17.6
	23-24	9.9	9.7-10.2	10.0
	24-25	10.9	10.2- 1.5	5.9
	25-26	16.6	1.5- 2.3	1.9
	26-27	2.7	2.3- 3.1	2.7
	27-28	23.2	3.1- 1.8	2.5
	28-29	55.9	1.8- 9.5	5.7
	29-30	36.0	9.5-12.7	11.1
	30-31	34.3	12.7- 5.1	8.9

(continued)

TABLE XXII. 39-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pits Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7504	1- 2	34.9	12.7- 6.4	9.6
VAPOR	2- 3	46.7	6.4- 5.8	6.1
	3- 4	27.6	5.8- 4.6	5.2
	4- 5	86.7	4.6-16.5	10.6
	5- 6	55.5	16.5- 2.0	9.3
	6- 7	8.4	2.0- 2.0	2.0
	7- 8	10.9	2.0- 3.6	2.8
	8- 9	78.2	3.6-23.9	13.8
	9-10	20.3	23.9- 7.1	15.5
	10-11	36.6	7.1- 7.6	7.4
	11-12	7.9	7.6- 3.6	5.6
	12-13	54.1	3.6-11.2	7.4
	13-14	79.5	11.2- 6.6	8.9
	14-15	16.0	6.6-18.3	12.5
	15-16	56.3	18.3- 9.7	14.0
	16-17	34.7	9.7- 4.8	7.3
	17-18	18.4	4.8-19.3	12.1
	18-19	48.3	19.3-76.2	47.8
	19-20	74.2	76.2-13.7	45.0
	20-21	111.9	13.7- 6.9	10.3
	21-22	32.0	6.9-11.4	9.2
	22-23	30.7	11.4- 6.9	9.2
	23-24	67.2	6.9-14.7	10.8
	24-25	46.2	14.7-15.7	15.2
7524	1- 2	44.1	55.1-25.4	40.3
LIQUID	2- 3	13.2	25.4-26.4	25.9
	3- 4	28.2	26.4-25.4	25.9
	4- 5	26.4	25.4-21.1	23.3
	5- 6	25.8	21.1-30.5	25.8
	6- 7	41.8	30.5-21.6	26.1

(continued)

TABLE XXII. 39-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pits Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7524	7- 8	48.9	21.6-25.4	23.5
LIQUID	8- 9	22.2	25.4-19.1	22.3
(Cont'd)	9-10	22.2	19.1-25.4	22.3
	10-11	31.5	25.4-17.3	21.4
	11-12	30.2	17.3-43.2	30.3
	12-13	56.5	43.2-19.1	31.2
	13-14	19.7	19.1-20.3	18.7
	14-15	31.8	20.3-25.4	22.9
	15-16	93.3	25.4-59.7	42.5
	16-17	54.6	59.7-34.3	47.0
	17-18	38.2	34.3-30.5	32.4
	18-19	63.5	30.5-96.5	63.5
	19-20	59.1	96.5-21.6	59.1
	20-21	40.0	21.6-45.7	33.7
	21-22	47.0	45.7-22.9	34.3
	22-23	43.2	22.9-50.8	36.9
	23-24	63.5	50.8-25.4	38.1
7524	1- 2	32.4	25.4-11.9	18.7
VAPOR	2- 3	15.6	11.9- 8.6	10.3
	3- 4	41.1	8.6-35.6	22.1
	4- 5	27.6	35.6- 5.3	20.5
	5- 6	28.1	5.3-38.1	21.7
	6- 7	31.8	38.1-25.4	31.8
	7- 8	44.5	25.4-33.0	29.2
	8- 9	48.3	33.0-38.10	35.6
	9-10	80.0	38.10-20.3	29.2
	10-11	24.8	20.3-11.4	15.9
	11-12	28.6	11.4-10.2	10.8
	12-13	44.7	10.2-28.4	19.3
	13-14	25.3	28.4-22.1	25.3

(continued)

TABLE XXII. 39-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pits Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7524	14-15	39.2	22.1-34.3	28.2
VAPOR	15-16	93.3	34.3-35.6	35.0
(Cont'd)	16-17	49.5	35.6-33.0	34.3
	17-18	41.9	33.0-30.5	30.3
	18-19	43.2	30.5-25.4	28.0
	19-20	36.7	25.4-21.6	23.5
	20-21	45.8	21.6-30.5	26.1
	21-22	49.7	30.5-38.1	34.3
	22-23	74.8	38.1-20.1	29.1
7556	1- 2	39.0	3.3- 3.6	3.5
LIQUID	2- 3	124.3	3.6- 3.8	3.7
	3- 4	131.2	3.8- 4.6	4.2
	4- 5	17.8	4.6- 3.0	3.8
	5- 6	102.1	3.0- 5.6	4.3
	6- 7	44.7	5.6-25.4	15.5
	7- 8	24.8	25.4- 1.3	13.4
	8- 9	73.5	1.3- 3.6	2.5
	9-10	9.1	3.6- 3.0	3.3
	10-11	14.9	3.0- 2.3	2.7
	11-12	127.9	2.3- 7.1	4.7
	12-13	159.8	7.1- 7.6	7.4
	13-14	132.1	7.6- 2.5	5.1
7556	1- 2	58.2	7.1- 7.6	7.4
VAPOR	2- 3	11.9	7.6-10.2	8.9
	3- 4	21.7	10.2-11.4	10.8
	4- 5	16.5	11.4-21.6	16.5
	5- 6	21.0	21.6-20.3	21.0
	6- 7	75.4	20.3- 8.6	14.5
	7- 8	11.9	8.6-15.2	11.9
	8- 9	26.4	15.2-25.4	20.3

(continued)

TABLE XXII. 39-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pits Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7556	9-10	17.1	25.4- 8.9	17.2
VAPOR	10-11	14.0	8.9-19.1	14.0
(Cont'd)	11-12	19.7	19.1-20.3	19.7
	12-13	18.4	20.3- 6.4	13.4
	13-14	76.8	6.4-15.2	10.8
	14-15	119.4	15.2-25.4	20.3
	15-16	341.6	25.4-17.8	21.6
	16-17	118.7	17.8-16.5	17.2
7574	1- 2	58.4	74.9-21.6	48.3
LIQUID	2- 3	82.6	21.6-62.2	41.9
	3- 4	54.0	62.2-45.7	54.0
	4- 5	81.9	45.7-67.3	56.5
	5- 6	48.1	67.3-19.8	43.6
	6- 7	35.3	19.8-50.8	35.3
	7- 8	40.6	50.8-30.5	40.7
	8- 9	27.9	30.5-25.4	28.0
	9-10	25.5	25.4-25.7	25.6
	10-11	25.5	25.7-25.4	25.6
	11-12	59.7	25.4-43.2	34.3
	12-13	40.0	43.2-19.1	31.2
	13-14	46.4	19.1-43.2	31.2
	14-15	128.3	43.2-71.1	57.2
	15-16	50.2	71.1-29.2	50.2
	16-17	57.8	29.2-50.8	40.0
	17-18	66.7	50.8-31.8	41.3
	18-19	57.2	31.8-82.6	57.2
	19-20	42.5	82.6- 2.5	42.6
	20-21	3.0	2.5- 3.6	3.1
	21-22	8.1	3.6-12.7	8.2

(continued)

TABLE XXII. 39-MONTH IMMERSION PIT SIZES AND LOCATIONS (Continued)

Coupon No. and Phase	Pits Nos.	Center-Center Pit Separation (cm x 10^{-4})	Diameter of Two Adjacent Pits (cm x 10^{-4})	Average Diameter of Two Adjacent Pits (cm x 10^{-4})
7574	1- 2	67.6	71.9-26.7	49.3
VAPOR	2- 3	41.0	26.7-14.7	20.7
	3- 4	39.8	14.7-14.0	14.4
	4- 5	25.4	14.0-19.1	16.6
	5- 6	16.8	19.1- 3.8	11.5
	6- 7	48.9	3.8-61.0	32.4
	7- 8	48.9	61.0-14.0	37.5
	8- 9	28.1	14.0-33.0	23.5
	9-10	26.7	33.0-20.3	26.7
	10-11	18.8	20.3-17.3	18.8
	11-12	42.9	17.3-25.4	21.4
	12-13	23.7	25.4-22.1	23.8
	13-14	45.1	22.1-68.1	45.1
	14-15	78.5	68.1-88.9	78.5
	15-16	95.3	88.9-101.6	95.3
	16-17	63.5	101.6-25.4	63.5
	17-18	54.6	25.4-61.0	43.2
	18-19	102.5	61.0-116.1	88.6
	19-20	87.9	116.1-29.2	72.7

TABLE XXIII. SUMMARY OF DIMENSIONS AND FREQUENCIES OF PITS IN 39-MONTH IMMERSION COUPONS

Coupon Number	Number of Pits Counted in 0.254 cm	Pit Frequency No. $x10^3/cm^2$	Pit Depth cm x 10^{-4}		Pit Area cm $x10^{-7}$		Depth/Diameter Ratio	
			Mean	Standard Dev.	Mean	Standard Dev.	Mean	Standard Dev.
7504-Liquid	78	94.3	2.08	7.16	33.80	77.29	0.31	0.31
7504-Vapor	63	61.5	1.52	5.39	28.01	89.86	0.23	0.16
7524-Liquid	60	55.8	3.88	1.56	109.26	150.34	0.13	0.06
7524-Vapor	58	52.1	5.61	3.37	74.88	59.11	0.22	0.15
7556-Liquid	35	19.0	1.14	0.47	4.91	13.24	0.33	0.28
7556-Vapor	43	28.7	1.36	7.16	20.94	15.73	0.10	0.06
7574-Liquid	55	46.9	6.47	3.30	153.55	157.09	0.25	0.22
7574-Vapor	50	38.8	4.17	3.23	216.19	300.85	0.13	0.09
Simple Mean	55.3	49.6	-	-	-	-	-	-
Weighted Mean	-	-	3.35	-	80.6	-	0.22	-
Range	35-78	19.0-94.3	1.14-6.47	-	4.91-216.19	-	0.10-0.33	-

TABLE XXIV. TENSILE MECHANICAL PROPERTIES OF 39-MONTH IMMERSION TITANIUM COUPONS

Coupon No.	Test Temp (K)	Ultimate Tensile Strength (psi)	Yield Strength (0.2% Offset) (psi)	% Elongation in 0.5 in.
7504-T	298	174,600	169,000	12
7504-B	77	255,600	-	10
7506-T	77	257,300	-	10
7506-B	298	194,600	162,300	14
7507-T	298	176,000	170,000	14
7507-B	298	196,900	170,000	12
7523-T	298	174,600	166,700	8
7523-B	77	255,500	-	10
7524-T	77	255,500	-	10
7524-B	298	194,500	172,000	10
7525-T	298	194,300	168,200	14
7525-B	298	174,200	168,000	10
7526-T	298	185,800	171,300	12
7526-B	298	176,100	169,300	10
7554-T	298	148,400	143,000	18
7554-B	298	147,700	142,600	16
7556-T	298	142,900	137,800	16
7556-B	298	145,700	142,900	16
7573-T	298	145,700	140,200	14
7573-B	298	148,000	140,600	14
7574-T	298	148,000	141,500	20
7574-B	298	150,800	143,100	18

NOTE: T = Top: Vapor Phase F_2 Immersion Exposure

B = Bottom: Liquid Phase F_2 Immersion Exposure

2.3.6 Analysis of Test Results and Observations

2.3.6.1 F_2 Composition/Chemical Analyses

Means and standard deviations for the residue analysis data (Tables XIV and XIX) were calculated, and are presented in Table XXV. In addition, the analyses of the starting F_2 (Table VII) were converted to the equivalent units, mg/capsule by the calculation:

weight of y, mg/capsule =

$$\frac{y\%\ v/v}{100} \times \frac{\text{Wt } F_2\text{, g/capsule}}{\text{Molecular Weight of } F_2} \times \text{Mol. Wt. of y} \times 1000 \text{ mg/g}$$

For the constituents reported as "CO_2 + HF", the assumption was made for the calculation that the material consisted of CO_2 only. The results are included in Table XXV. The data indicate, first, that there still are problems in obtaining meaningful analytical data from systems which contain elemental fluorine. The 29-month control test (no titanium) demonstrates this, when compared with initial concentrations and other test sequences.

The much lower impurity contents from the tests compared to the initial fluorine are due to the purification by distillation which occurs when less volatile materials remained in the graduated cylinder during initial fills - - see Section 2.3.4.1(a), paragraph (vi).

The SiF_4 concentration determination is difficult to evaluate. Although the 39 month controls have significant concentrations in all samples, only 3 out of 15 of the titanium-compatibility capsules gave appreciable concentrations; the other 12 were below the detectable limit. It may be that SiF_4 was produced inside those capsules which have measurable amounts, perhaps by reaction between water, fluorine, and glass, if the capsules were incompletely dried or had picked up water during manipulation.

The CO_2 content seems to be about constant, within statistical significance bands.

The content of HF, which is the most corrosive of the impurities which were measured, and of CF_4, are below the detectability limit in all measurements.

TABLE XXV. COMPARISON OF ANALYSES FOR VOLATILE IMPURITIES IN FLUORINE (mg PER CAPSULE)

	SiF_4			CO_2			HF	CF_4
	$\overline{X}$	n	σ	$\overline{X}$	n	σ	$\overline{X}$	$\overline{X}$
Pre-Test*	<36	1	-	7.2**	1	-		<42
29-Mo. Control	<0.01	2	-	<0.01	2	-	<0.04	<0.01
39-Mo. Control	0.223	4	0.171	0.433	4	0.335	<0.04	<0.01
29-Mo. Titanium	0.025 (0.1)	4 (1)	0.05 (--)	0.305	4	0.172	<0.04	<0.01
39-Mo. Titanium	0.068 (0.38)	11 (2)	0.152 (0.035)	0.265	10	0.075	<0.04	<0.01

* Calculated for 15.5g of F_2 from data in Table VII.

** Based on assumtion that "CO_2 + HF" is all CO_2.

2.3.6.2 Visual, Microscopic and Electron-microscope Examinations

Discoloration and pitting were observed for both the 29 months and 39 months exposure periods. There was no visual distinction between the two exposure periods.

2.3.6.3 Metallography, Pitting Analysis, and Weight Changes

The technique used to prepare the coupons for metallography and pitting analysis includes immersion in an acid aqueous solution. Because titanium fluorides are soluble in or decomposed by water, this step probably removed most of the corrosion products, but the unreacted titanium surface should not have been affected. Therefore the metallography and pitting studies are believed to apply to the condition of the substrate metal, and not to the condition of a coating of corrosion product film.

Metallography has not revealed the development of any significant change of mode of attack by the fluorine.

Accumulated statistical data for the pre-exposure and post exposure periods were derived from the statistical results where the mean values were determined from the following equation:

$$\text{MEAN } \overline{X} = \Sigma \frac{\overline{X}_1 \times N_1 + \overline{X}_2 \times N_2 \ . \ .}{\Sigma N}$$

Where: $\overline{X}$ = Mean is the total average of all specimens with the same parameters.

$\overline{x}$ = Average of values for one specimen.

N_1, N_2 = Number of readings from each specimen.

N = Total number of sets of $\overline{X}_1 \overline{X}_2 \overline{X}_3 \overline{X}_4$ averages.

This treatment of the data was used to generate Tables XXVI and XXVII for pit dimensions and for pit frequencies, respectively.

TABLE XXVI. SUMMARY OF PIT DIMENSIONS

Exposure Time/ Fluorine Phase	Condition/ Surface Finish	Pit Depth cm x 10^{-4}	Pit Area cm^2 x 10^{-7}	Depth/Diameter Ratio
None	A/16	1.35	0.88	0.27
29 Months/Liquid	A/16	1.58	24.70	0.27
39 Months/Liquid	A/16	1.14	4.91	0.33
29 Months/Vapor	A/16	1.74	1.10	0.58
39 Months/Vapor	A/16	1.36	20.94	0.10
29 Months/All	A/16	1.64	16.01	0.38
39 Months/All	A/16	1.26	13.75	0.20
None	A/64	1.74	1.09	0.83
29 Months/Liquid	A/64	5.45	15.86	0.59
39 Months/Liquid	A/64	6.47	153.55	0.25
29 Months/Vapor	A/64	6.30	21.30	0.76
39 Months/Vapor	A/64	4.17	216.19	0.13
29 Months/All	A/64	5.99	19.29	0.70
39 Months/All	A/64	5.38	183.40	0.19
None	A/ALL	1.41	0.95	0.45
29 Months/All	A/ALL	5.16	18.67	0.64
39 Months/All	A/ALL	3.62	121.10	0.19
None	H/16	1.07	1.37	0.52
29 Months/Liquid	H/16	2.06	4.59	0.31
39 Months/Liquid	H/16	2.08	33.80	0.31
29 Months/Vapor	H/16	2.11	4.44	0.35
39 Months/Vapor	H/16	1.52	28.01	0.23
29 Months/All	H/16	2.08	4.53	0.33
39 Months/All	H/16	1.83	31.21	0.27
None	H/64	1.72	1.52	0.85
29 Months/Liquid	H/64	1	1	[illegible]
39 Months/Liquid	H/64	3.88	109.26	0.13
29 Months/Vapor	H/64	6.02	20.00	0.38
39 Months/Vapor	H/64	5.61	74.88	0.22
29 Months/All	H/64	6.02	20.00	0.38
39 Months/All	H/64	4.73	92.36	0.17
None	H/ALL	1.30	1.42	0.64
29 Months/All	H/ALL	3.27	9.20	0.35
39 Months/All	H/ALL	3.15	59.07	0.22
None	ALL/ALL	1.35	1.23	0.56
29 Months/All	ALL/ALL	4.51	15.39	0.54
39 Months/All	ALL/ALL	2.76	80.61	0.21

1 Not Tested

TABLE XXVII. SUMMARY OF PIT FREQUENCY PER UNIT AREA

Exposure Time/ Fluorine Phase	Condition/ Surface Finish	Measured Number of Pits in 0.25 cm	Calculated Pit Frequency 10^3 Number Pits/cm^2
None	A/16	9.5	1.55
29 Months/Liquid	A/16	12.0	2.20
39 Months/Liquid	A/16	35.0	19.00
29 Months/Vapor	A/16	7.0	0.80
39 Months/Vapor	A/16	43.0	28.70
None	A/64	9.0	1.30
29 Months/Liquid	A/64	19.0	5.60
39 Months/Liquid	A/64	55.0	46.90
29 Months/Vapor	A/64	34.0	17.90
39 Months/Vapor	A/64	50.0	38.80
29 Months/All	A/16	9.5	1.50
39 Months/All	A/16	39.0	23.85
29 Months/All	A/64	26.5	11.75
39 Months/All	A/64	52.5	42.85
None	A/ALL	9.3	1.47
29 Months/Liquid	A/ALL	15.5	3.90
39 Months/Liquid	A/ALL	45.0	32.95
29 Months/Vapor	A/ALL	20.5	9.35
39 Months/Vapor	A/ALL	46.5	33.75
29 Months/All	A/ALL	17.6	6.10
39 Months/All	A/ALL	45.8	33.35
None	H/16	13.0	2.73
29 Months/Liquid	H/16	11.5	2.05
39 Months/Liquid	H/16	78.0	94.30
29 Months/Vapor	H/16	7.0	0.85
39 Months/Vapor	H/16	63.0	61.50
None	H/64	14.5	3.45
29 Months/Liquid	H/64	11.0	1.90
39 Months/Liquid	H/64	60.0	55.80
29 Months/Vapor	H/64	16.5	4.25
39 Months/Vapor	H/64	58.0	52.10
29 Months/All	H/16	9.3	1.45
39 Months/All	H/16	70.5	77.90
29 Months/All	H/64	14.7	3.47
39 Months/All	H/64	59.0	53.95
None	H/ALL	13.5	2.97
29 Months/Liquid	H/ALL	11.3	2.00
39 Months/Liquid	H/ALL	69.0	75.05
29 Months/Vapor	H/ALL	11.8	2.55
39 Months/Vapor	H/ALL	60.5	56.80
29 Months/All	H/ALL	11.6	2.31
39 Months/All	H/ALL	64.8	65.93

Comparison of the pitting analysis results for the 3 classes of exposure shows a considerable change in the character of the metal surface. Combined data from all tests on pit dimensions is presented in Table XXVI and on pit frequencies in Table XXVII. As would be expected, pit depth, pit area, and pit frequency are greater for the 64 RMS finish than for the 16 RMS finish. Data on the average character of the surfaces is presented in Table XXVIII. Review of the average surface data shows a significant, accelerating increase both in the number of pits and in the average area of the pits. The number of pits doubles in 29 months and then is multiplied by 10 in the next 10 months, about 20-fold overall; the average area is multiplied by 13 in the first period and by 6 in the second, about 75-fold over the whole test.

The average pit area and the number of pits per unit area (Table XXVII) are plotted against the immersion period in Figure 10. These graphs emphasize the increase over the final ten month period. A plot of the total pit volume per unit area (penetration rate) is given as a function of time in Figure 11. The notable characteristic is not the absolute penetration rate at 39 months, 4.5×10^{-5} cm/year, but rather its acceleration from 6.5×10^{-6} cm/year at 29 months, an increase of 7 times.

On the other hand, the change in average pit depth is only about 2-fold, and apparently levels off. In addition, there is an approximate correspondence between the number of pits on the pre-exposure coupons and the number of pits appreciably deeper than average on the 39 month coupons. The penetration of these deeper pits may be of significance for longer exposures. The maximum pit depths measured, on 64 RMS coupons, are shown in Table XXIX.

The weight changes shown in Table XV and XXI and combined in Table XXX represent an average weight gain of 0.31%, relative to the initial weights of the complete specimens. After 39 months, the weight gain has dropped to 0.13%. The decrease is shown by all 4 conditions of the specimens. The probability bands are independent at the 1σ level, but they overlap at the 2σ level of significance.

TABLE XXVIII. CHANGES IN PIT SIZES AND DISTRIBUTION

	Pre-Immersion	29-Month	39-Month
Center-center spacing, cm	2.2×10^{-2}	1.8×10^{-2}	4.6×10^{-3}
Number per cm^2	2.2×10^{-3}	3.9×10^{3}	49.6×10^{3}
Average depth, cm	1.34×10^{-4}	4.50×10^{-4}	3.35×10^{-4}
Average area, cm^2	1.23×10^{-7}	15.4×10^{-7}	80.6×10^{-7}
Average depth/diameter ratio	0.67	0.54	0.22
Total pit volume* per unit area (cm^3/cm^2) = Average depth of material removed, cm	3.36×10^{-8}	2.70×10^{-6}	1.34×10^{-4}

*Assumes cylindrical shape: V = area x depth x number.

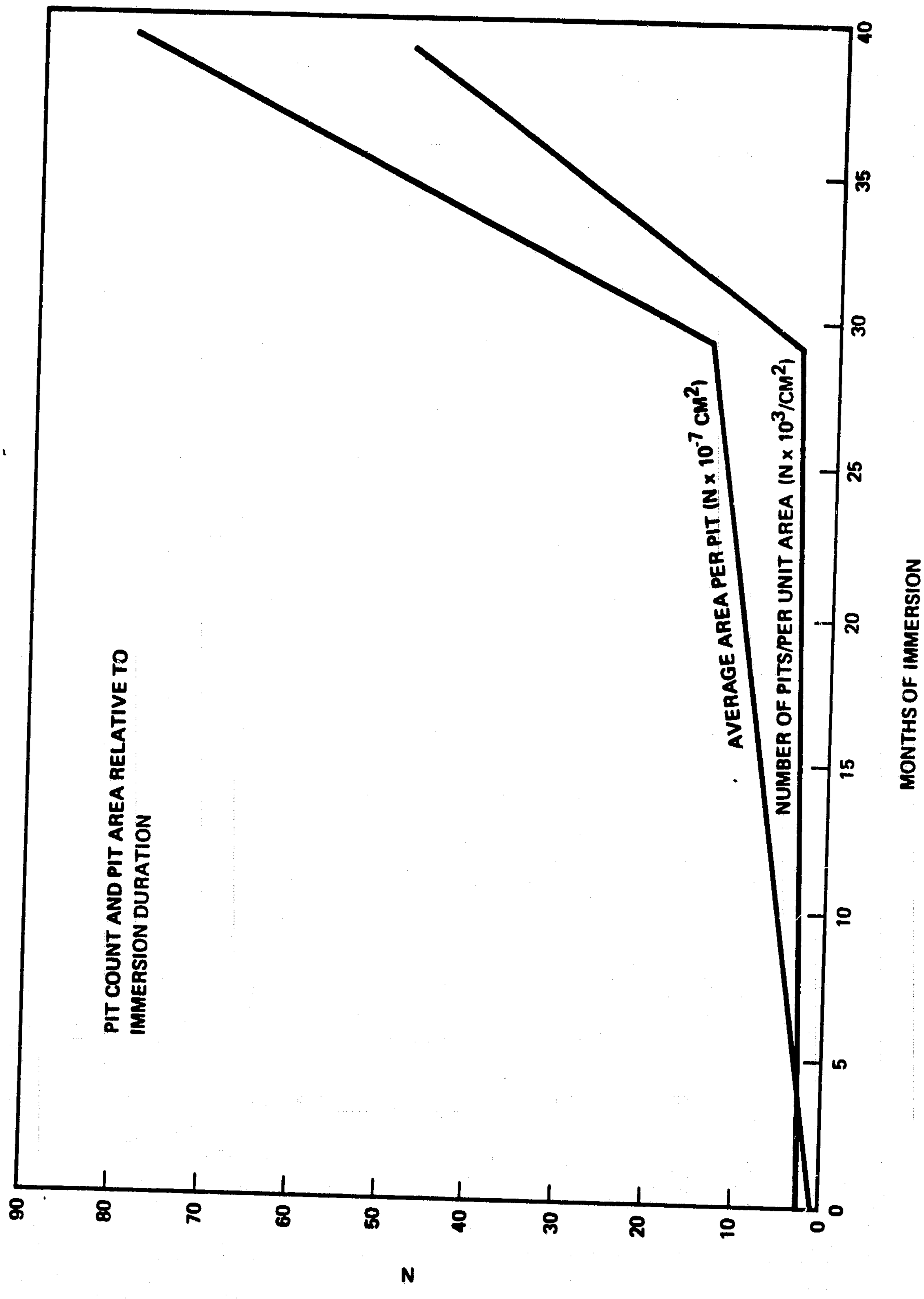

Figure 10. Variation of Pitting with Time

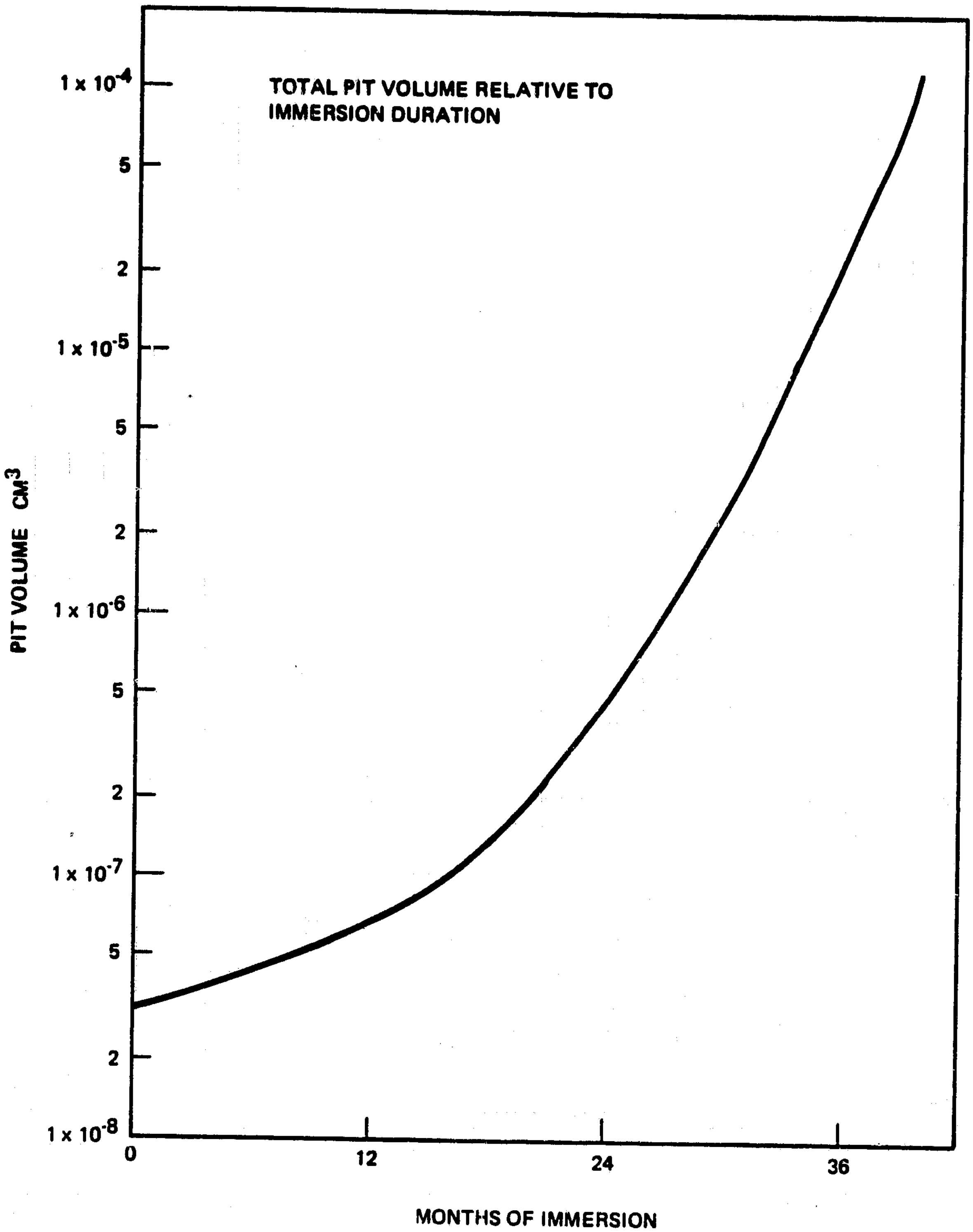

Figure 11. Variation of Volume of Titanium Removed with Time

TABLE XXIX. MAXIMUM PIT DEPTHS

Coupon	Condition	Test Category	Depth(cm)
7520	H-64	Pre	0.42×10^{-3}
7570	A-64	Pre	0.42
7527-L	H-64	29 Mo.	1.8
7527-V	H-64	29 Mo.	2.1
7572-V	A-64	29 Mo.	2.0
7575-L	A-64	29 Mo.	0.89
7575-V	A-64	29 Mo.	1.1
7524-L	H-64	39 Mo.	0.89
7524-V	H-64	39 Mo.	1.5
7574-L	A-64	39 Mo.	1.5
7574-V	A-64	39 Mo.	1.5

TABLE XXX. WEIGHT CHANGE VERSUS EXPOSURE TIME

Exposure Time	Condition/ Surface Finish	Average Weight Gain (%)
29 Months	A/16	0.35
39 Months	A/16	0.21
29 Months	A/64	0.42
39 Months	A/64	0.08
29 Months	A/ALL	0.38
39 Months	A/ALL	0.13
29 Months	H/16	0.29
39 Months	H/16	0.09
29 Months	H/64	0.37
39 Months	H/64	0.16
29 Months	H/ALL	0.33
39 Months	H/ALL	0.13
29 Months	ALL/ALL	0.35
39 Months	ALL/ALL	0.13

Data from this test was compared to those obtained from a 14 month exposure period (Reference 4). The pit frequency from the 14 month exposure was 4 to 9 X 10^4 pits/cm^2, approximately the same as the pit frequency found after the 39 month exposure (1.9 to 9.4 X 10^4 pits/cm^2), but the 29 month exposure had a much lower pit frequency (8 to 34.7 X 10^2 pits/cm^2). Pit areas appeared to be smaller after the 14 months of exposure: 1 X 10^{-8} to 6 X 10^{-6} cm^2/14 months; 1.5 X 10^{-6} cm^2/29 months (average); 8.1 X 10^{-6} cm^2/36 months (average). The maximum pit depth increase markedly after the 14 months exposure: 8 X 10^{-4} cm for 14 months; 2 x 10^{-3} cm for 29 months; and, 1.5 X 10^{-3} cm for 36 months.

In the weight changes measured after 29 months, it is noted that annealed material of 64 RMS finish shows a statistically significant greater weight gain (95% probability) than the other materials. This is not the case for the 39 month tests; while all materials are indistinguishable statistically, and the A-64 specimens showed the lowest mean gain. Recall the discussion in Section 2.3.5.4(d); it is probable that the weight change data are not independent of post-test handling and hence have little predictive value.

2.3.6.4 Mechanical Properties (Tensile)

A summary of all the tensile testing is presented in Table XXXI. The test data show that the mechanical properties of the titanium specimens when tested at either 298 K or 77 K are not degraded by the liquid/gas fluorine exposure. It should be noted that this testing at the slow strain rate cannot be used to predict the strength of the titanium at appreciably faster strain rates (i.e., impact loading). The pits in the titanium have increased in depth and area and the larger pits may lower the impact strength.

2.3.7 Discussion of Results

The compatibility tests described in this report were conducted with fluorine which had been subjected to cryogenic single-plate (pot) distillation. This occurred because of the technique used to fill the test capsules. The purity of the fluorine was probably approximately that recorded for control samples in Table XXV, and not the pre-test material. The very slow rate of attack by the fluorine on the alloy coupons is in accord with this assumption.

Variations in the attack on the alloy do not correlate very significantly with the fluorine phase, nor with the condition of the Ti 6AL-4V. The best correlation is between pit development and exposure duration. However, there is appreciable variability from capsule to capsule which share the same test conditions. From general familiarity with fluorine compatibility, it is believed that these variations reflect differences in the initial condition of the specimens and capsules when the fluorine was introduced. The prime variable suspected in trace water content. Although the assemblies were treated similarly, there was no effort to attain identical histories, and the initial water content (absorbed on metal and glass) was not measured.

Because the test data indicate that corrosion rates do not level off in the 3 year test period, the compatibility/corrosion rates for longer periods cannot be predicted. The lack of leveling off may be related to the apparent fragility of thicker films of fluoride. This may turn out to be significant for longer exposures.

The severity of pitting corrosion of Ti 6 AL-4V in F_2 appears to increase with exposure duration. However, the currently available data are not sufficient for calculation of corrosion rates and design life for contact in excess of 3 years.

Because the observed corrosion effects involves pit formation and pit depth increase, the behavior of F_2-exposed Ti-6AL-4V under dynamic loads is unknown and cannot be predicted from currently available data.

2.4 APPLICATION IN DESIGN

For the design of a specific LF_2 system, selection of structural materials must be based on knowledge of various modes of interaction between the fluorine and the materials under all system operation modes and on evaluation of how such interactions affect the performance of the system. The user should consult the NASA reports "Fluorine Systems Handbook," NASA CR-72064, July 1, 1967 and "Experience With Fluorine and Its Safe Use as a Propellant" by D. L. Bond et al, JPL Publication 79-64, June 30, 1979, before using the data in this document.

TABLE XXXI. SUMMARY OF TENSILE MECHANICAL PROPERTY DATA

Exposure Time/ Fluorine Phase	Condition2/ (Heat Treatment/ Surface Finish)	Test Temp.	Ultimate Tensile Strength (KSI)			Yield Strength (KSI)			Elongation (1/2 Inch Gage Length)		
			Range	Mean	Standard Deviation	Range	Mean	Standard Deviation	Range	Mean	Standard Deviation
0	A/16	298 K	146.7 to 147.3	147.1	0.32	138.2 to 139.6	138.9	0.70	0	16.0	0
29 Months/Liquid	A/16	298 K	1/	146.2	1/	1/	139.1	1/	1/	20.0	1/
39 Months/Liquid	A/16	298 K	145.7 to 147.7	146.7	1.41	142.6 to 142.9	142.8	0.21	0	16.0	0
29 Months/Vapor	A/16	298 K	1/	150.4	1/	1/	147.2	1/	1/	16.0	1/
39 Months/Vapor	A/16	298 K	142.9 to 148.4	145.7	3.89	137.8 to 143.0	140.4	3.68	16 to 18	17.0	1.41
0	A/64	298 K	1/	146.5	1/	1/	139.1	1/	1/	16.0	1/
29 Months/Liquid	A/64	298 K	1/	149.2	1/	1/	141.3	1/	1/	18.0	1/
39 Months/Liquid	A/64	298 K	148.0 to 150.8	149.4	1.98	140.6 to 143.1	141.9	1.77	14 to 18	16.0	2.83
29 Months/Vapor	A/64	298 K	1/	149.6	1/	1/	140.1	1/	1/	18.0	1/
39 Months/Vapor	A/64	298 K	145.7 to 148.1	146.9	1.70	140.2 to 141.5	140.9	0.92	14 to 20	17.0	4.24
0	H/16	298 K	175.0 to 176.6	175.8	0.80	161.9 to 165.2	163.5	1.66	10 to 12	10.7	1.15
29 Months/Liquid	H/16	298 K	173.1 to 174.4	173.8	0.92	163.5 to 165.1	164.3	1.13	14 to 16	15.0	1.41
39 Months/Liquid	H/16	298 K	194.6 to 196.9	195.8	1.63	162.3 to 170.0	166.2	5.44	12 to 14	13.0	1.41
29 Months/Vapor	H/16	298 K	177.0 to 177.8	177.4	0.57	166.5 to 167.9	167.2	0.99	0	16.0	0
39 Months/Vapor	H/16	298 K	174.6 to 176.0	175.3	0.99	169.0 to 170.0	169.5	0.71	12 to 14	13.0	1.41
0	H/64	298 K	1/	173.8	1/	1/	159.4	1/	1/	13.0	1/
29 Months/Liquid	H/64	298 K	175.2 to 177.0	176.1	1.27	165.2 to 166.2	165.7	0.71	0	16.0	0
39 Months/Liquid	H/64	298 K	174.2 to 194.3	181.5	11.10	168.0 to 172.0	169.8	2.04	0	10.0	0
29 Months/Vapor	H/64	298 K	174.6 to 177.5	176.1	2.05	164.3 to 166.8	165.6	1.77	0	14.0	0
39 Months/Vapor	H/64	298 K	174.6 to 194.3	184.9	9.88	166.7 to 171.3	168.7	2.35	8 to 12	10.0	2.0
0	A/16	77 K	225.8 to 231.7	228.8	4.17	3/	3/	3/	8 to 9	8.5	0.71
29 Months/Liquid	A/16	77 K	1/	221.5	1/	3/	3/	3/	1/	12.0	1/
39 Months/Liquid	A/16	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
29 Months/Vapor	A/16	77 K	4/	4/	4/	3/	3/	3/	4/	4/	4/
39 Months/Vapor	A/16	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
0	A/64	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
29 Months/Liquid	A/64	77 K	1/	224.0	1/	3/	3/	3/	1/	7.0	1/
39 Months/Liquid	A/64	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
29 Months/Vapor	A/64	77 K	1/	250.0	1/	3/	3/	3/	1/	6.0	1/
39 Months/Vapor	A/64	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
0	H/16	77 K	256.0 to 256.4	256.2	0.28	3/	3/	3/	0	6.0	0
29 Months/Liquid	H/16	77 K	1/	244.4	1/	3/	3/	3/	1/	8.0	1/
39 Months/Liquid	H/16	77 K	1/	255.6	1/	3/	3/	3/	1/	10.0	1/
29 Months/Vapor	H/16	77 K	4/	4/	4/	3/	3/	3/	4/	4/	4/
39 Months/Vapor	H/16	77 K	1/	257.3	1/	3/	3/	3/	1/	10.0	1/
0	H/64	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
29 Months/Liquid	H/64	77 K	1/	254.2	1/	3/	3/	3/	1/	5.0	1/
39 Months/Liquid	H/64	77 K	1/	255.5	1/	3/	3/	3/	1/	10.0	1/
29 Months/Vapor	H/64	77 K	1/	251.1	1/	3/	3/	3/	1/	5.0	1/
39 Months/Vapor	H/64	77 K	1/	255.5	1/	3/	3/	3/	1/	10.0	1/
0	A/ALL	298 K	146.5 to 147.3	146.9	0.39	138.2 to 139.6	139.0	0.58	0	16.0	0
29 Months/Liquid	A/ALL	298 K	146.2 to 146.5	146.4	0.21	139.1 to 141.3	140.2	1.56	0	16.0	0
39 Months/Liquid	A/ALL	298 K	145.7 to 150.8	148.0	2.10	140.6 to 143.1	142.3	1.15	14 to 18	16.0	1.63
29 Months/Vapor	A/ALL	298 K	149.6 to 150.4	150.0	0.57	140.1 to 147.2	143.7	5.02	16 to 18	17.0	1.41
39 Months/Vapor	A/ALL	298 K	142.9 to 148.4	146.3	2.55	137.8 to 143.0	140.6	2.20	14 to 20	17.0	2.58
29 Months/ALL	A/ALL	298 K	146.2 to 150.4	148.2	2.14	139.1 to 147.2	141.9	3.63	16 to 18	16.5	1.00
39 Months/ALL	A/ALL	298 K	142.9 to 150.8	147.2	2.36	137.8 to 143.1	141.5	1.86	14 to 20	16.5	2.07
0	H/ALL	298 K	173.8 to 176.6	175.1	1.40	159.4 to 165.2	162.6	2.96	10 to 13	11.7	1.53
29 Months/Liquid	H/ALL	298 K	173.1 to 177.0	174.9	1.63	163.5 to 166.2	165.0	1.12	14 to 16	15.5	1.00
39 Months/Liquid	H/ALL	298 K	174.2 to 196.9	187.3	11.12	162.3 to 172.0	168.3	3.66	10 to 14	11.2	1.79
29 Months/Vapor	H/ALL	298 K	174.6 to 177.8	176.7	1.45	164.3 to 166.8	166.4	1.51	14 to 16	15.0	1.15
39 Months/Vapor	H/ALL	298 K	176.0 to 194.3	181.1	8.76	166.7 to 171.3	169.0	1.75	8 to 14	12.0	2.45
29 Months/ALL	H/ALL	298 K	173.1 to 177.8	175.8	1.72	163.5 to 166.8	165.7	1.43	14 to 16	15.25	1.04
39 Months/ALL	H/ALL	298 K	174.2 to 196.9	184.2	9.98	162.3 to 172.0	168.7	2.73	8 to 14	11.6	2.07
0	A/ALL	77 K	225.8 to 231.7	228.8	4.17	3/	3/	3/	8 to 9	8.5	0.71
29 Months/Liquid	A/ALL	77 K	221.5 to 224.0	222.8	1.77	3/	3/	3/	7 to 12	9.5	3.54
39 Months/Liquid	A/ALL	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
29 Months/Vapor	A/ALL	77 K	1/	250.0	1/	3/	3/	3/	1/	6.0	1/
39 Months/Vapor	A/ALL	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
29 Months/ALL	A/ALL	77 K	221.5 to 250.0	231.9	15.78	3/	3/	3/	6 to 12	8.3	3.21
39 Months/ALL	A/ALL	77 K	3/	3/	3/	3/	3/	3/	3/	3/	3/
0	H/ALL	77 K	256.0 to 256.4	256.2	0.28	3/	3/	3/	0	6.0	0
29 Months/Liquid	H/ALL	77 K	244.4 to 254.2	249.3	6.93	3/	3/	3/	5 to 8	6.5	2.12
39 Months/Liquid	H/ALL	77 K	255.5 to 255.6	255.6	0.07	3/	3/	3/	0	10.0	0
29 Months/Vapor	H/ALL	77 K	1/	251.0	1/	3/	3/	3/	1/	5.0	1/
39 Months/Vapor	H/ALL	77 K	255.5 to 257.3	256.4	1.27	3/	3/	3/	0	10.0	0
29 Months/ALL	H/ALL	77 K	244.4 to 254.2	249.9	5.00	3/	3/	3/	5 to 8	6.0	1.73
39 Months/ALL	H/ALL	77 K	255.5 to 257.3	256.0	0.88	3/	3/	3/	0	10.0	0

1/ One specimen
2/ A - Annealed
H - Heat treat hardened
3/ Not tested
4/ Specimen broke in holding fixture

3. CONCLUSIONS

- Based on the immersion test results for up to 3 years, titanium 6A1-4V is compatible with fluorine at 77 K for periods of as long as 3 years.
- Titanium 6AL-4V with an initally smoother surface is more resistant to pitting than the same alloy with a rougher surface.
- Because the test data indicate that corrosion rates do not level off in the 3 year period, the compatibility for longer periods cannot be predicted.
- Because the observed corrosion mechanism involves pit formation and pit depth increase, the behavior of the F_2-exposed titanium under dynamic loads is unknown and cannot be predicted.
- The tensile strength and ductility of the 6AL-4V titanium alloy, when tested at a slow strain rate, have not been degraded by the three year exposure to the vapor and liquid fluorine.
- There appears to be no significant difference between the corrosion resistance of annealed and heat treated titanium.
- The 64 RMS specimens appear to corrode at a rate approximately three times that of the 16 RMS specimens.
- The size and number of pits increases with time, but there is insufficient data to determine the corrosion rate function.

- As the pits continue to increase in number and size with exposure to the vapor and liquid fluorine, there may be a significant drop in the titanium fracture toughness.

- There is a possibility that long duration exposure to F_2 results in fragile corrosion films which can crack away and result in significant solid particle contamination of the F_2.

4. RECOMMENDATIONS

(1) It is recommended that fracture toughness testing of Ti-6AL-4V be conducted in liquid and vapor fluorine environments at contemplated storage temperature and stress conditions. The increase in pitting severity with time may eventually lead to development of critical crack depth thereby resulting in catastrophic fracture at stress level below the yield strength of the material.

(2) It is recommended that additional tests including longer terms exposures be conducted to provide data which can be used for developing a corrosion rate equation.

(3) It is recommended that design curves for long term storage of Ti-6AL-4V alloy in F_2 environments be generated using fracture toughness data and corrosion rate data. The design curves should include life expectancy projection based on stress levels, F_2 conditions, temperature, and alloy conditions.

(4) It is recommended that the strength and structure of fluoride corrosion films on titanium be studied as a function of exposure time.

New Technology

No reportable items of new technology have been identified.

REFERENCES

1. F. Champion, "Corrosion Testing Procedures," John Wiley and Sons, N. Y., 1956.

2. 4343.3.76-098, "Input to JPL Contract 954450 Summary Report", A. Toy to J.R. Denson, 10 June 1976.

3. 5515.2.79-103, "Post Immersion Analyses of Titanium Specimens-Tankabe", J. Roth to J.R. Denson, 22 February, 1979.

4. L.L. Constantino, J.R. Denson, C.S. Krishnan and A. Toy, "Compatibility Testing of Spacecraft Materials and Space-Storable Liquid Propellants", TRW Report 23162-6023-RU-00, May 1976, prepared for JPL on Contract 953486.

APPENDIX A

TITANIUM 6AL-4V
CERTIFICATION AND ANALYSIS

FUTURA TITANIUM CORPORATION

RA691

SOLD TO:	SHIP TO: P/O REF 613425 C-55872

ITEM	CUSTOMER ORDER NO.	OUR ORDER NO.	DATE SHIPPED	DESCRIPTION	SIZE

CHEMICAL ANALYSIS

HEAT #	C	FE	N_2	AL	VA	CR	MO	H_2	O_2	MN	TI	SN	NI	ELEMENTS

PHYSICAL PROPERTIES

HEAT #	YIELD STRENGTH PSI	TENSILE STRENGTH PSI	ELONG % IN 2"	RED. OF AREA %	HARDNESS	REMARKS	TEST #

SWORN TO AND SUBSCRIBED BEFORE ME

THIS________DAY OF________________, 19____

____
NOTARY PUBLIC

CERTIFICATE OF TEST

THE TEST RESULTS SHOWN IN THIS REPORT ARE CORRECT TO THE BEST OF OUR KNOWLEDGE AND BELIEF.

FUTURA TITANIUM CORPORATION

CERTIFIED BY:____________________________
REPRESENTATIVE

FTSC—4 NCR—TR—2500—7/73

TITANIUM SUPPLY

15915 So. San Pedro Street, Gardena, California 90248 (213) 532-6371

SALES ORDER

80-359

CUSTOMER ORDER NUMBER

GT 620932

528 1 5 1974 80-359

AONO4 04037 80 001 10220532 TS 08
8006

P/O 613425
C-35572

Jet Propulsion Lab
California Institute of Tech.
4800 Oak Grove Drive
Pasadena, CA 91103

WILL CALL

CERTIFICATE OF TEST
NOTICE OF SHIPMENT

6AL/4V Titanium to MIL-T 9046 F Type 3 Comp C Ann.

	INVOICE NUMBER	DATE
	564 224	May 29, 1974

VIA	GROSS	TARE	NET
WILLCALL			2 1/2 lbs.

ITEM	DESCRIPTION	NO. PCS.	SQUARE FEET	BILLING WT. PER PIECE OR SQ. FT.	WEIGHT			BAL. DUE
1	.032 x RW x RL Heat Number K 8800 Test Number L 8283	2	2		2 1/2			C

CERTIFIED CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES
ON REVERSE SIDE

TITANIUM SUPPLY

CERTIFICATE OF TEST

CHEMICAL ANALYSIS

HEAT NO.	C	FE	N	AL	VA	CR	MO	H	ZR	SN	MN	O
K 8800	.026	.09	.012	6.0	4.1			.011				.10

MECHANICAL PROPERTIES

HEAT NO.	TEST NO.	SIZE OR GAUGE	.2% YIELD STRENGTH	TENSILE STRENGTH	ELONG.	R. A. %	HARDNESS	BEND TEST
		.032	KSI	KSI				R/T @ 20X
K 8800	L 9293	L	147	157	11			4.0
		T	138	152	10			4.0
		L	150	161	11			4.0
		T	130	149	10			4.0
			MFG: TIMET					

SUBSCRIBED AND SWORN TO BEFORE ME

THIS DATE May 29, 1974

RESULTS AS ABOVE CERTIFIED

TITANIUM SUPPLY GARDENA CALIFORNIA

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APPENDIX B

TITANIUM 6AL-4V SPECIMENS

HEAT TREAT AND SURFACE FINISH METHOD, CERTIFICATION, AND TEST

C - 2

PACIFIC STEEL TREATING CO. PST

A 26733

6829 FARMDALE AVE. • NORTH HOLLYWOOD, CALIF. 91605
983-1450 877-3346

WT.

CUSTOMER: JET PROPULSION LABORATORY
PROC. ENG.:
DATE: 6-7-4
P.O.: NT 613434
JOB DESCRIPTION: 20 STRIPS .032 X 1/16" X 6"
SHIPPER: L-85777
CUST. JOB NO.: 778-RA691-1-2320
CONTAINERS: REF. P/O 613425 C-85272
A/C PRIME:

MATERIAL: 6AL-4V TI
CONDITION: AGED
SURFACE FINISH: CAST, WROUGHT ☐ ROUGH MACH ☐ FINISH MACH ☒
MAX SECTION THICKNESS: .032

HARDNESS REQUIREMENTS — ROCKWELL: — BRINELL: — CORE: — 100% ☐ PARTIAL ☐ SPECIMEN ☐

TENSILE REQUIREMENTS — U/T: — KSI YIELD: — KSI ELONG: — % R.A.: — % NO. COUPONS TO PULL:

NO.	OPERATION	SPECIFICATION	MEDIUM	TEMP.	REQ'D SOAK TIME	FURN. NO.	DATE	TIME IN	TIME AT TEMP	TIME OUT	STAMP
1	VAC PURGE RETORT							A.M. P.M.	A.M. P.M.	A.M. P.M.	PST 40
2	ANNEAL	MIL-H 81200A	AR	1425	30 MIN	43	6/8	12:10 P.M.	12:30 P.M.	12:50 P.M.	PST 40
3	RETORT COOL TO R/T							A.M. P.M.	A.M. P.M.	A.M. P.M.	PST 40

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3
TESTS

ACTUAL HARDNESS — ROCKWELL: BRINELL: CORE:

AUTHORIZED SIGNATURE: [signature]

TITLE: a/28 RC-2191

JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

INSPECTION REPORT

I. R. NO. A 1914

SHEET 1 OF 1

MO.	DAY	YR
8	13	75

PROGRAM	REF. DESIG.	S/N	NOMENCLATURE	P.O.	REQ. NO.
306	UK	NA	DOGBONE	NA	NA

	SUB ASSY	ACCT. CODE NO.	REG. NO.	CONTRACT NO.
NA	NA -306	12103-0-53	0*0063	NA

OPERATION AFFECTED	QTY. REC.		CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER	SUPPLIER
DIM & FINISH	19										
DWG/PART NO.: 10056227-1 · REV.: B · CONFIG.: NA		REQ.	Yes							Yes	JPL
WHERE INSP. PERFORMED: SOURCE (JPL) SAF ETR · MRB NO.:		FURN.	NO							No	

COG. ENG.	EXT.	LEVEL OF INSP.	TYPE HARDWARE	TRAVELER NO.
TOTH	3225	SHIP · REC. · (IN-PROC.) · TEST · FINAL	FLT. · PROTO · TA · SE	NA

STATUS	SPEC. NO.	TYPE	LOT NO.	INSP. LEVEL CHK.
NA	NA	NA	NA	100%

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1		.0025/IN FLATNESS CK'S .005/.009	UAI	use as is	
2		.030±.001 DIM. CK'S .032/.034	UAI	use as is	
3		NO MAT'L CERTS WITH PARTS	UAI	use as is	
		NOTE: PENDING IDENT & PASSIVATION PER DWG.			

INSPECTED BY: W Templin (JPL 84)

SIGN, STAMP, DATE: W Templin 8/13/75 (JPL 84)

ITEM	SUPPLEMENTARY INFO./CORRECTIVE ACTION
	All use as-is R. Wilson

QTY. ACCP'T.	QTY. REJ.	PARTS REC. BY
19	0	[illegible]

COG. ENG.	DATE	Q. A. ENG.	DATE
L Toth			8-18-75

JPL 1453 R 2/75

ACCOMPANY HARDWARE

jpl → **MECHANICAL FABRICATION INSPECTION REQUEST**

				REG. NO.
				Q. NO.
DATE 8-19-75	ENGINEER L. TOTH			P.O. NO.
REQUESTED BY L. TOTH	BLDG. 125	ROOM 224	EXT. 3215	I.R. NO. A 3413
DRAWING NO.	QUANTITY		SERIAL NO.	~~PROJECT NO.~~ 9-4-75
TITLE OR DESCRIPTION METALLIC SPECIMEN, DOUBLE DOGBONE				ACC. NO. 306-12103-0-3840

DETAILED INSTRUCTIONS, SKETCH, ETC.

Inspect parts per attached drawing dated 8-19-75 and as follows:

1. Ten (10) parts
   compliance with $\stackrel{16}{\surd}$ finish
2. Six (6) parts
   compliance with $\stackrel{64}{\surd}$ finish

Notes
1. Cross reference IR No. A 1914
2. Material is 6AL-4V Titanium, annealed condition. Notch on end of part is used to identify annealed condition (reference note 8 on dwg).
3. Determine typical surface finish of each part, and note value on each envelope

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OF POOR QUALITY

DELIVER TO	BLDG.	ROOM	DATE REQUIRED
P. WILLSON	125	224	8-29-75

Q.A. FILE

or sooner

JPL JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

INSPECTION REPORT

I. R. NO. A 3413

SHEET 1 OF 1 | MO. 9 | DAY 4 | YR 75

PROGRAM	REF. DESIG.	S/N	NOMENCLATURE	P.O.	REQ. NO.
306	NA	NA	Metallic Specimen	NA	NA

	SUB ASSY	ACCT. CODE NO.	REG. NO.	CONTRACT NO.
R&D	Specimen	306-12103-0-3840	Q 30915	NA

OPERATION AFFECTED	QTY. REC.		CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER	SUPPLIER
Dim. Insp.	16	REQ.		NA							JPL

DWG/PART NO.	REV.	CONFIG.
10056727	B	NA

WHERE INSP. PERFORMED: SOURCE | JPL | SAF | ETR | MRB NO.

FURN. NA

COG. ENG.	EXT.	LEVEL OF INSP.	TYPE HARDWARE	TRAVELER NO.
L. Toth	3225	SHIP / REC. / IN-PROC. / TEST / FINAL	FLT. / PROTO / TA / SE	NA

STATUS	SPEC. NO.	TYPE	LOT NO.	INSP. LEVEL CHK.
NA	NA	NA	NA	100%

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1		.030±.001 Thickness is up to			
		.032 to .033 MAX.			
2		Flat within .025/in. is .016			
		one specimen (#3)			
3		No material certification			
4		No Passivation certification			
		Note:		L. TOTH	
		(1) Assigned test No's pending		9-4-75	
		(2) See actual dims recorded			
		on each envelope.			

INSPECTED BY: Joe Bulaki (JPL 115)

SIGN, STAMP, DATE

ITEM	SUPPLEMENTARY INFO./CORRECTIVE ACTION
	Note: These units are not for flight
	L. TOTH 9-4-75

QTY. ACCP'T.	QTY. REJ.	PARTS REC. BY
16		

COG. ENG.	DATE	Q. A. ENG.	DATE
L. TOTH	9-4-75		

ACCOMPANY HARDWARE

JPL JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
4800 OAK GROVE DRIVE PASADENA, CALIFORNIA

INSPECTION REPORT

I. R. NO. [illegible]

SHEET 1 OF 1

MO. [illegible] DAY [illegible] YR 7[illegible]

PROGRAM: [illegible]
REF. DESIG.: [illegible]K
S/N: N/A
NOMENCLATURE: [illegible]
P.O.: [illegible]
REQ. NO.: [illegible]

R & D
SUB ASSY: N/A
ACCT. CODE NO.: [illegible]
REQ. NO.: [illegible]
CONTRACT NO.: N/A

OPERATION AFFECTED: DIM. INSP.
QTY. REC.: [illegible]

	CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER
REQ.		YES	YES					
FURN.		YES	YES					

SUPPLIER: [illegible]

DWG/PART NO.: [illegible]-1-B
MRB NO.: N/A
MRB ITEM NO.:

WHERE INSP. PERFORMED: SOURCE | JPL | SAF | AMR

COG. ENG.: [illegible]
EXT.: 7177
LEVEL OF INSP.: REC. | IN-PROC. | TEST | FINAL
TYPE HARDWARE: FLT. | PROTO | TA | OSE
TRAVELER NO.:

STATUS: N/A
SPEC. NO.: N/A
TYPE: N/A
LOT NO.: N/A
INSP. LEVEL CHK.: [illegible]

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1.		MAT'L FAILS .270		[illegible]	
		.289 ON 30 ANNEALED		[illegible]	
		SPECIMENS.			
2.		MAT'L FAILS [illegible]			
		.287, 14 PCS, NOT		[illegible]	
		ANNEALED.			
		NOTE:			
		[illegible]			
		PENDING.			

INSP. [illegible] (JPL 280)
DATE [illegible] (JPL 57)

ITEM	CORRECTIVE ACTION
	R/AD item
	Not for flight purposes -

QTY. ACCP'T.:
QTY. REJ.:
PARTS REC. BY:

COG. ENG.: [illegible]TH
DATE: 7-18-7[illegible]
Q. A. ENG.:
DATE:

JPL 1453 (2-68)

ACCOMPANY HARDWARE

MECHANICAL FABRICATION INSPECTION REQUEST

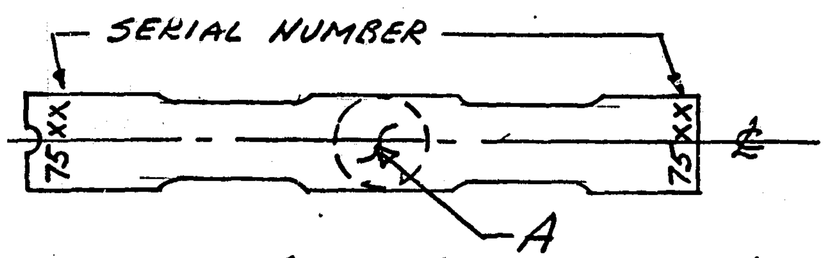
jpl

REG. NO.	
Q. NO.	Q30918
P.O. NO.	
I.R. NO.	
PROJECT NO.	R/D
ACC. NO.	306-12103-0-3840

DATE	ENGINEER		
FEB 23, 1976	L. TOTH		
REQUESTED BY	**BLDG.**	**ROOM**	**EXT.**
L. TOTH	125	224	3225
DRAWING NO.	**QUANTITY**	**SERIAL NO.**	
10056227B	5	NOTED	

TITLE OR DESCRIPTION
Metallic Specimen Double Dogbone
(special parts - annealed condition)

DETAILED INSTRUCTIONS, SKETCH, ETC.



Determine surface finish on both sides of specimen in center portion, A, and record below:

Serial Number	I.D. Side rms	Opposite Side rms
7560	12 RMS	14 RMS
7561	17 RMS	15 RMS
7562	15 RMS	14 RMS
7576	42 RMS	42 RMS
7577	41 RMS	41 RMS

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DELIVER TO	BLDG.	ROOM	DATE REQUIRED
P. Willson or L. TOTH	125	224	MAR 2, 1976 or sooner

Q.A. FILE

JPL 2726 R2/71

JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

INSPECTION REPORT

I. R. NO. A 13137

SHEET 1 OF 1

MO.	DAY	YR
2	25	76

PROGRAM	REF. DESIG.	S/N	NOMENCLATURE	P.O.	REQ. NO.
DL	11K	NOTED * BELOW	SPECIMEN DOUBLE-DOGBONE	NA	NA

	SUB ASSY	ACCT. CODE NO.	REG. NO.	CONTRACT NO.
NA	SPECIMEN DOUBLE -DOGBONE	306-1103-0-3840	930910	NA

OPERATION AFFECTED: Surface Finish Insp.
QTY. REC.: 5

	CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER
REQ.	NA							
FURN.	NA							

DWG/PART NO.: 10056227 REV.: B CONFIG.: NA

WHERE INSP. PERFORMED: SOURCE (JPL) SAF ETR

MRB NO.: NA

SUPPLIER: J.P.L.

COG. ENG.: L. TOTH EXT.: 3225

LEVEL OF INSP.: SHIP REC. (IN-PROC.) TEST FINAL

TYPE HARDWARE: FLT. PROTO TA SE

TRAVELER NO.: NA

STATUS: NA SPEC. NO.: NA TYPE: NA LOT NO.: NA INSP. LEVEL CHK.: 100%

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1	/	3 ALL SURFACES 16 TO 8. S/N 7576 Finish is (42 RMS) BOTH SIDES. S/N 7577 Finish is (41 RMS) BOTH SIDES.		Special parts used for R/AD application. Finish acceptable as is.	
		* SURFACE FINISH INSPECTION ONLY ON S/N's 7560, 7561, 7562, 7576 AND 7577. FOR RMS READ OUTS SEE ATTACHED SHEET.		L. TOTH	

INSPECTED BY: Kenneth Lowe (JPL 130)

SIGN, STAMP, DATE:

ITEM	SUPPLEMENTARY INFO./CORRECTIVE ACTION
	Parts used for R/AD program

QTY. ACCP'T.: 5 QTY. REJ.: 0 PARTS REC. BY:

COG. ENG.: Louis R. Toth DATE: 3-3-76 Q. A. ENG.: DATE:

ACCOMPANY HARDWARE

JPL 1453 R 2/75

JPL

FABRICATION WORK ORDER REQUEST

ORIGINATOR: COMPLETE UN-SHADED AREAS. REFER TO SPI 6-01-80

ACCOUNT NO. TO BE CHARGED	WORK ORDER NUMBER
3 0 2 4 2 6 0 2 0 3 8 4 0	RA 691

WORK ORDER STATUS (19)

N OPEN NEW W.O.	T OPEN/COMPL NEW W.O.	A INCREASE PRIOR ESTIMATE
B DECREASE PRIOR ESTIMATE	M ADD NEW ACTIVITY	OTHER

DATE	PROGRAM OR PROJECT		
5-14-74			
REQUESTER'S NAME	BLDG/ROOM	EXT	
L. Toth	125-224	3225	
DELIVER MATERIAL TO	CERT OF MAT'L REQ'D	DATE REQUIRED	
P. Willson	X YES ☐ NO	6-21-74 or sooner	
DRAWING NO	QTY	DRAWING TITLE	
10056227B	32	Metallic Specimen	
REQUESTER'S ESTIMATE			
$ 1,600.00			

RPT (20)	DOING SECT.	LOCATION	EST. COMPL. DATE
N	7 3 0		
DATE THIS INPUT	LIAISON MAN		
JOB TITLE			

INSTRUCTIONS

Provide the following samples made from 6AL-4V titanium material per Mil-T-9046.

1. Material: Annealed condition

   A. Fabricate eight specimens per dwg. 10056227-1.
   B. Fabricate eight specimens per dwg. 10056227-1 except that all surfaces to have a 64√ finish.
   C. Provide a 12" X 12" piece of parent material.

2. Material: Heat treated

   A. Fabricate eight specimens per dwg. 10056227-1.
   B. Fabricate eight specimens per dwg. 10056227-1 except that all surfaces to have a 64√ finish.
   C. Provide a 12" X 12" piece of parent material.

3. Notes

   (1) Any cleaning or surface preparation required shall be done only with isopropyl alcohol. Do not use Freon under any circumstances.
   (2) Dwg. 10056227B notes 4, 5, 6, and 7: Not part of this order; to be handled by cognizant engineer.

ESTIMATED COST

CODE (48)							TOTALS
LABOR	HOURS (49-55)						HOURS
	DOLLARS (56-61)						$
OUTSIDE CONTRACT (62-67)							$
MATERIAL AND OTHER (68-73)							$
IN-HOUSE SUPPORT (74-79)							$

REQUIRED APPROVALS —— TOTAL ESTIMATE → $ 74 79

ORIGINATOR	GROUP SUPERVISOR	DATE	SECTION MANAGER	DATE	DIVISION MANAGER	DATE
L. Toth	L. Toth	5-16-74				
PROGRAM DIRECTOR	DATE	ASSISTANT LABORATORY DIRECTOR	DATE	DIRECTOR/DEPUTY DIRECTOR	DATE	

FABRICATION WORK ORDER REQUEST

ORIGINATOR: COMPLETE UN-SHADED AREAS
REFER TO SPI 6-01-60

ACCOUNT NO. TO BE CHARGED	WORK ORDER NUMBER
30612103 03840	CM 122

DATE	PROGRAM OR PROJECT		
8-7-75	R/AD		
REQUESTER'S NAME		BLDG/ROOM	EXT.
L. TOTH		125/224	3225

N OPEN NEW W.O.	T OPEN COMPL. W.O.	A INCREASE ORDER ESTIMATE
B DECREASE ORDER ESTIMATE	M ADD NEW ACTIVITY	OTHER

DELIVER MATERIAL TO	CERT. OF MAT L. REQ'D.	DATE REQUIRED
L. TOTH	☐ YES ☒ NO	8-29-75

	SUBJECT	LOCATION	EST. COMPL. DATE
N	7 3 0		

DRAWING NO	QTY	DRAWING TITLE
10056227B		Specimens - double dog bone

DATE TO SHOP	LIAISON MAN

REQUESTER'S ESTIMATE
$ 99.00

JOB TITLE

INSTRUCTIONS

Hand polish specimens as follows:

1. Polish eleven (11) specimens on all surfaces using crocus cloth to meet a 16 finish

2. Polish eight (8) specimens on all surfaces to meet a 64 finish

Notes:

1. Test coupon material: 6AL-4V titanium heat treated condition
2. Polish specimens in dry condition.
3. Do not use any freon type solvents

ESTIMATED COST

CODE (48)							TOTALS
LABOR	HOURS (49-55)						HOURS
	DOLLARS (56-61)						$
OUTSIDE CONTRACT (62-67)							$
MATERIAL AND OTHER (68-73)							$
IN HOUSE SUPPORT (74-79)							$

REQUIRED APPROVALS → TOTAL ESTIMATE $

ORIGINATOR	GROUP SUPERVISOR	DATE	SECTION MANAGER	DATE	DIVISION MANAGER	DATE
L. TOTH						
PROGRAM DIRECTOR		DATE	ASSISTANT LABORATORY DIRECTOR	DATE	DIRECTOR/DEPUTY DIRECTOR	DATE

FABRICATION WORK ORDER REQUEST
ORIGINATOR: COMPLETE UN-SHADED AREAS
REFER TO SPI 6-01-60

ACCOUNT NO. TO BE CHARGED: 306 / 12 / 03 / 03840

RT211

PROGRAM OR PROJECT: R/AD

NAME: L. TOTH

BLDG/ROOM: 125/224

EXT: 3225

DELIVER MATERIAL TO: L. TOTH

CERT. OF MAT'L. REQ'D. ☒ YES ☐ NO

DATE REQUIRED: 8-15-75

N 7 3 0

DRAWING NO: 1[illegible]7B

QTY: 4

DRAWING TITLE: metal specimen double dog bone

LIAISON MAN

ESTIMATE: $ 25.00

INSTRUCTIONS

Heat treat four (4) material compatibility test specimens to full heat treat condition.

Note
1. Material is 6AL-4V titanium, annealed condition
2. Test specimens are in finished condition.

ORIGINAL PAGE IS OF POOR QUALITY

ESTIMATED COST

CODE (49)						TOTALS
LABOR HOURS (49 55)						HOURS
LABOR DOLLARS (56 61)						$
OUTSIDE CONTRACT (62 67)						$
MATERIAL AND OTHER (68 73)						$
IN HOUSE SUPPORT (74 79)						$

REQUIRED APPROVALS —— TOTAL ESTIMATE → $ 74

ORIGINATOR	GROUP SUPERVISOR	DATE	SECTION MANAGER	DATE	DIVISION MANAGER	DATE
L. TOTH						

PROGRAM DIRECTOR	DATE	ASSISTANT LABORATORY DIRECTOR	DATE	DIRECTOR/DEPUTY DIRECTOR	DATE

FABRICATION WORK ORDER REQUEST

ORIGINATOR: COMPLETE UNSHADED AREAS
REFER TO SPI 6-01-60

ACCOUNT NO. TO BE CHARGED	WORK ORDER NUMBER
306 121030 3840	CM130

DATE	PROGRAM OR PROJECT		
9-11-75	R/AD		
REQUESTER'S NAME	BLDG/ROOM	EXT.	
L. TOTH	125/224	3225	
DELIVER MATERIAL TO	CERT. OF MAT'L REQ'D.	DATE REQUIRED	
L. TOTH	☐ YES ☒ NO	9-2-75 or sooner	
DRAWING NO.	QTY	DRAWING TITLE	
10056227B	16	Specimens - double dog bone	
REQUESTER'S ESTIMATE			
$ 87.00			

N	OPEN NEW W.O.	T	OPEN COMPL. NEW W.O.	A	INCREASE PRIOR EST'MATE
B	DECREASE PRIOR ESTIMATE	M	ADD NEW ACTIVITY		OTHER

	FUNDING SECT	LOCATION	EST. COMPL. DATE
N	7 3 0		

LIAISON MAN

PART TITLE

INSTRUCTIONS

Hand polish specimens as follows:

1. Polish ten (10) specimens on all surfaces using crocus cloth to meet a $\sqrt[16]{}$ finish.
2. Polish six (6) specimens on all surfaces to meet a $\sqrt[64]{}$ finish

Notes:

1. Test coupon material: 6AL-4V Titanium annealed condition. Parts are identified with "A" in left hand corner
2. Polish specimens in dry condition.
3. Do not use any freon type solvents.

ESTIMATED COST

CODE (48)						TOTALS
LABOR HOURS (49-55)						HOURS
LABOR DOLLARS (56-61)						$
OUTSIDE CONTRACT (62-67)						$
MATERIAL AND OTHER (68-73)						$
IN HOUSE REPORT						$

REQUIRED APPROVALS → TOTAL ESTIMATE $

ORIGINATOR	GROUP SUPERVISOR	DATE	SECTION MANAGER	DATE	DIVISION MANAGER	DATE
L. TOTH						
PROGRAM DIRECTOR	DATE	ASSISTANT LABORATORY DIRECTOR	DATE	DIRECTOR/DEPUTY DIRECTOR	DATE	

jpl → MECHANICAL FABRICATION INSPECTION REQUEST

DATE 8-14-75	ENGINEER L. TOTH			REG. NO. / Q. NO. / P.O. NO.
REQUESTED BY L. TOTH	BLDG. 125	ROOM 224	EXT. 3225	I.R. NO.
DRAWING NO. 10056227B	QUANTITY NOTED		SERIAL NO.	PROJECT NO.
TITLE OR DESCRIPTION METALLIC SPECIMEN, DOUBLE DOGBONE				ACC. NO. 306-12103-0-3840

DETAILED INSTRUCTIONS, SKETCH, ETC.

Inspect surface finish as follows:

1. Eleven (11) parts
   Compliance with $\sqrt{63}$ finish

2. Eight (8) parts
   Compliance with $\sqrt{64}$ finish

Note

a. If practical, determine typical surface finish of each part, and note value on each envelope.

b. Material is 6AL-4V Titanium
   Full heat treat condition

ORIGINAL PAGE IS
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DELIVER TO	BLDG.	ROOM	DATE REQUIRED
P. WILLSON	125	224	8-20-75

Q.A. FILE

JPL 2726 R2/

jpl

FABRICATION WORK ORDER REQUEST

ORIGINATOR: COMPLETE UN-SHADED AREAS.
REFER TO SPI 6-01-60

ACCOUNT NO. TO BE CHARGED (1–13): 306 12103 03840

WORK ORDER NUMBER (14–19): RT231

WORK ORDER STATUS (11): N — OPEN NEW W.O. | T — OPEN/COMPL NEW W.O. | A — INCREASE PRIOR ESTIMATE | B — DECREASE PRIOR ESTIMATE | M — ADD NEW ACTIVITY | OTHER

RPT (20)	DO NB SECT. (26–29)	LOCATION (30–34)	EST. COMPL. DATE (35–40)
N	7 3 0		

DATE THIS INPUT (41–46):

LIAISON MAN:

JOB TITLE (47–64):

DATE: 8-19-75

PROGRAM OR PROJECT: R/D

REQUESTER'S NAME: L. TOTH

BLDG/ROOM: 125/224

EXT: 3225

DELIVER MATERIAL TO: P. Willson

CERT. OF MAT'L. REQ'D.: ☐ YES ☒ NO

DATE REQUIRED: 9-3-75

DRAWING NO: 10056127B

QTY: 12

DESCRIPTION: Metallic specimen [illegible] double dog bone

REQUESTER'S ESTIMATE: $ 150.00

INSTRUCTIONS

Fabricate twelve (12) parts per dwg 10056227 B.

Notes:

1. Material furnished is 6AL-4V Titanium STA heat treat condition per specification MIL-T-9046F
2. Return residual material to JPL
3. Do not use any Freon type solvents for processing. Machine dry if possible and use ~~[illegible]~~ isopropyl alcohol for cleaning.

ESTIMATED COST

CODE (48)							TOTALS
LABOR — HOURS (49-55)							HOURS
LABOR — DOLLARS (56-61)							$
OUTSIDE CONTRACT (62-67)							$
MATERIAL AND OTHER (68-73)							$
IN-HOUSE SUPPORT 74-79							$

REQUIRED APPROVALS → TOTAL ESTIMATE → $ (74–79)

ORIGINATOR	GROUP SUPERVISOR	DATE	SECTION MANAGER	DATE	DIVISION MANAGER	DATE
L. TOTH						

PROGRAM DIRECTOR	DATE	ASSISTANT LABORATORY DIRECTOR	DATE	DIRECTOR/DEPUTY DIRECTOR	DATE

jpl → **MECHANICAL FABRICATION INSPECTION REQUEST**

REG. NO.
Q. NO.

DATE	ENGINEER			P.O. NO.
8-19-75	L. TOTH			
REQUESTED BY	BLDG.	ROOM	EXT.	I.R. NO.
L. TOTH	125	224	3225	A 3413
DRAWING NO.	QUANTITY		SERIAL NO.	~~PROJECT NO.~~
				9-4-75
TITLE OR DESCRIPTION				ACC. NO.
METALLIC SPECIMEN, DOUBLE DOGBONE				306-12103-0-3840

DETAILED INSTRUCTIONS, SKETCH, ETC.

Inspect parts per attached drawing dated 8-19-75 and as follows:

1. Ten (10) parts
   Compliance with $\sqrt{}$16 finish
2. Six (6) parts
   Compliance with $\sqrt{}$64 finish

Notes
1. Cross reference IR No. A 1914
2. Material is 6AL-4V Titanium, annealed condition. Notch on end of part is used to identify annealed condition (reference note 8 on dwg).
3. Determine typical surface finish of each part, and note value on each envelope

DELIVER TO	BLDG.	ROOM	DATE REQUIRED
P. WILLSON	125	224	8-29-75

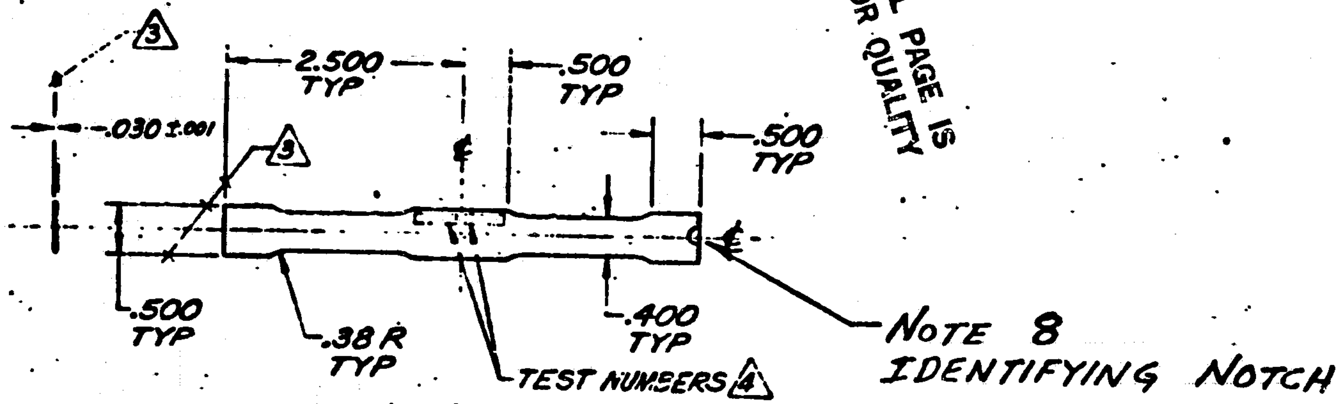
Q.A. FILE

or sooner

JPL 2726 R2/71

ORIGINAL PAGE IS OF POOR QUALITY

LTR	DESCRIPTION	DATE	APPROVED
A	INITIAL RELEASE		
B	ADDED ITEM 19 TO PARTS LIST		2-1-77



8: TEST SAMPLES WITH NOTCH
MATERIAL 6AL-4V TITANIUM
ANNEALED CONDITION, MIL-T-9046F

7. TOTAL SURFACE AREA = 4.73 SQ IN
VOLUME = 0.066 CU IN.
6. IDENTIFY WITH PART NO. PER JPL SPEC FS500451; PARA. 3.5.4.
5. CLEAN AND PASSIVATE PER JPL SPEC FS506300.
4. TEST NUMBERS TO BE ASSIGNED BY JPL CCG ENGR.
3. ALL SURFACES 16/ TO 8/.
2. ALL SURFACES FLAT WITHIN .0025/IN.
1. REMOVE ALL BURRS AND SHARP EDGES .005 MAX.

NOTES: UNLESS OTHERWISE SPECIFIED

ITEM NO.	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	SPECIFICATION	MATERIAL
19	-19	SPECIMEN	MIL-A-8920	2219-T87 AL ALY
18	-18			TUNG CBD
17	-17		QQ-A-250/11	6061-T6 AL ALY
16	-16		MIL-A-8920	2219-T81
15	-15		QQ-A-250/4	2024-T3
14	-14		QQ-A-250/1	1100 AL ALY
13	-13		MIL-S-25043	17 7PH CRES
12	-12		AMS 5604	17-4PH
11	-11		QQ-S-763	416
10	-10		QQ-S-763	430
9	-9		MIL-S-8840	AM355
8	-8		MIL-S-8840	AM350
7	-7		MIL-S-6721	321
6	-6		MIL-S-5059	316L
5	-5		MIL-S-4043	304L
4	-4		MIL-S-7720	303
3	-3		MIL-S-5059	302
2	-2		MIL-S-6721	347 CRES
1	10056227-1	SPECIMEN	MIL-T-9046	6AL-4V TI

PARTS LIST

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES
TOLERANCES
DECIMALS ANGLES
MACHINE FINISH 3.2
DO NOT SCALE DRAWING

MATERIAL
SEE PARTS LIST

R&D

JET PROPULSION LABORATORY
356

METALLIC SPECIMEN, DOUBLE - DOGBONE

C | 23835 | 10056227 | B

8-19-75 / L. TOTH

C 10056227

MECHANICAL FABRICATION INSPECTION REQUEST

DATE 9-1-75	ENGINEER L. TOTH			REG. NO.
				Q. NO.
				P.O. NO.
REQUESTED BY L. TOTH	BLDG. 125	ROOM 224	EXT. 3225	I.R. NO.
DRAWING NO. 1005 6227 B	QUANTITY 15		SERIAL NO.	PROJECT NO.
TITLE OR DESCRIPTION Metallic Specimen, Double Dogbone				ACC. NO. 306-12103-0-3890

DETAILED INSTRUCTIONS, SKETCH, ETC.

Inspect parts for dimensional compliance.

Notes

1. Material is 6AL-4V titanium, heat treated condition

2. Surface finish is probably out of print and will be polished later.

3. Material certification - not required material furnished by JPL

DELIVER TO P. Willson	BLDG. 125	ROOM 229	DATE REQUIRED 9-8-75

Q.A. FILE

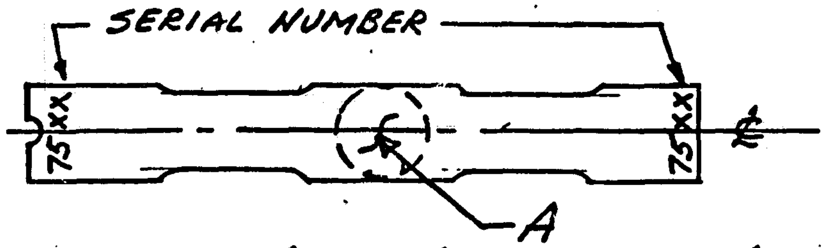
a. [illegible]

JPL 2726 R2/71

MECHANICAL FABRICATION INSPECTION REQUEST

REQ. NO.			
Q. NO. 030918			
DATE FEB 23, 1976	ENGINEER L. TOTH		P.O. NO.
REQUESTED BY L. TOTH	BLDG. 125 / ROOM 224	EXT. 3225	I.R. NO.
DRAWING NO. 10056227B	QUANTITY 5	SERIAL NO. NOTED	PROJECT NO. R/D
TITLE OR DESCRIPTION Metallic Specimen Double Dogbone (special parts - annealed condition)			ACC. NO. 306-12103-0-3840

DETAILED INSTRUCTIONS, SKETCH, ETC.



Determine surface finish on both sides of specimen in center portion, A, and record below:

Serial Number	I.D. Side rms	Opposite Side rms
7560	12 RMS	14 RMS
7561	17 RMS	15 RMS
7562	15 RMS	14 RMS
1576	42 RMS	42 RMS
7577	41 RMS	41 RMS

ORIGINAL PAGE IS OF POOR QUALITY

DELIVER TO	BLDG.	ROOM	DATE REQUIRED
P. Willson or L. TOTH	125	224	MAR 2, 1976 or sooner

Q.A. FILE

JPL 2726 R2/71

JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

INSPECTION REPORT

I. R. NO. A 1816?

SHEET 1 OF 1

MO.	DAY	YR
[illegible]	[illegible]	[illegible]

PROGRAM	REF. DESIG.	S/N	NOMENCLATURE	P.O.	REQ. NO.
[illegible]	NA	[illegible]	[illegible]	NA	[illegible]

BY	SUB ASSY	ACCT. CODE NO.	REG. NO.	CONTRACT NO.
NA	SPECIMEN [illegible]	[illegible]	[illegible]	NA

OPERATION AFFECTED	QTY. REC.		CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER	SUPPLIER
Surface Finish [illegible]	5	REQ.	NA								JPL
		FURN.	NA								

DWG/PART NO.: [illegible] REV.: [illegible] CONFIG.: NA

WHERE INSP. PERFORMED: SOURCE | JPL | SAF | ETR — MRB NO.: NA

COG. ENG.: L. Toth EXT.: [illegible]

LEVEL OF INSP.: SHIP | REC. | IN-PROC. | TEST | FINAL

TYPE HARDWARE: FLT. | PROTO | TA | SE

TRAVELER NO.: NA

STATUS	SPEC. NO.	TYPE	LOT NO.	INSP. LEVEL CHK.
NA	NA	NA	NA	[illegible]

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1	/	3 All surfaces 16/ to 8/ S/N 7576 Finish is (43 RMS) both sides. S/N 7577 Finish is (41 RMS) both sides.		[illegible] R/AD [illegible]	
				F[illegible]	
		* Surface Finish Inspection only on S/N 7560, 7561, 7562 7576 and 7577. For RMS readings see attached sheet.		L. Toth	

INSPECTED BY: [illegible] (JPL 138)

SIGN, STAMP, DATE:

ITEM	SUPPLEMENTARY INFO./CORRECTIVE ACTION
	Only used for R/AD program

ORIGINAL PAGE IS OF POOR QUALITY

QTY. ACCP'T.	QTY. REJ.	PARTS REC. BY
5	0	

COG. ENG.	DATE	Q. A. ENG.	DATE
[illegible] Toth	3-3-76		

ACCOMPANY HARDWARE

JPL 1453 R 2/75

jpl **TEST REQUEST**

76-1500-
PAGE NO. ____ OF ____

ITEM Metallic Specimen double dogbone
DWG. NO. 10056227B REV. ____
SPEC. NO. FS504574, cleaning spec. REV. C
JPL S/N Noted VENDOR P/N ____ S/N ____

- [x] CLEAN ROOM
- [ ] DATA SHEET
- [ ] INSTRUMENTATION
- [ ] OVERTIME OK
- [ ] PHOTOGRAPHY
- [ ] Q/A REQUIRED
- [ ] WITNESS REQD
- [ ]
- [ ] DEVELOPMENT
- [ ] FUNCTIONAL
- [ ] FLIGHT ACCEPTANCE
- [ ] TYPE APPROVAL

REQUESTER L. TOTH EXT 3225 DATE REQUIRED Sept 10, 1976
ALTERNATE ____ EXT. ____ DATE OF REQUEST Aug 12, 1976

JOB NUMBER 306-12103-0-3440
START OF TEST ____ COMP ____ CLOSE ____
EST HOURS ____ ACT HRS ____ OT ONLY ____
PROJECT SSPM
RECD BY Wm Douglas DATE 8/16/76
APPROVED ____ DATE ____

INSTRUCTIONS

1. Please clean titanium test samples in accordance with specification FS504574C, Tables II and III to a D-2 level and in the sequence noted on

   TR 76-0600 (copy attached.) items 1, 2, and 3

2. Specimens have been returned from TRW, contract 954450, task 1 work.

3. Packaging - Bag each set similar to "as received" since serial number does not appear on all pieces.

4. Serial numbers are

Specimens tested			Specimens examined	
7500	R.T	(1/2)	7500	(1)
7502	R.T	(2/4)	7501	(2)
7503	-320°F	(2/4)	7520	(1)
7520	R.T.	(1/2)	7521	(2)
7550	R.T.	(1/2)	7550	(1)
7552	R.T.	(2/4)	7551	(2)
7553	-320°F	(2/4)	7570	(1)
7570	R.T.	(1/2)	7571	(2)

Completed 8/24/76 Wm Douglas

ORIGINATOR

JPL 3224 (REV. 12-69)

TEST REQUEST

76-0601-
PAGE NO. 1 OF 1

ITEM Metallic Specimen Double Dogbone
DWG. NO. 10056227B REV.
SPEC. NO. FS504574 Cleaning Spec. REV. C
JPL S/N Noted VENDOR P/N S/N

- [x] CLEAN ROOM
- [ ] DATA SHEET
- [ ] INSTRUMENTATION
- [ ] OVERTIME OK
- [ ] PHOTOGRAPHY
- [ ] Q/A REQUIRED
- [ ] WITNESS REQD
- [ ]
- [ ] DEVELOPMENT
- [ ] FUNCTIONAL
- [ ] FLIGHT ACCEPTANCE
- [ ] TYPE APPROVAL

REQUESTER L. TOTH EXT. 3225 DATE REQUIRED 3-24-76
ALTERNATE EXT. DATE OF REQUEST 3-8-76

INSTRUCTIONS

1. Please clean five (5) titanium test samples in accordance with instructions listed on TR 76-0600 (copy attached)
2. Package separately; leave history card separate from specimens.
3. Serial numbers are

S/N 7560 Surface finish 16√
7561 "
7562 "
7576 Surface finish 64√
7577 "

JOB NUMBER 306-12103-0-3840
START OF TEST
EST HOURS
COMP
ACT HRS
CLOSE
OT ONLY
PROJECT SSPM
RECD BY
DATE
APPROVED
DATE

ORIGINATOR

JPL 3224 (REV. 12-69)

TITANIUM SUPPLY

RA691

15915 So. San Pedro Street, Gardena, California 90248 (213) 532-6371

SALES ORDER

80-359

CUSTOMER ORDER NUMBER AND DATE: CT 620932 — 528 1 5 1974

NUMBER: 80-359

A0N04 04037 80 001 10220532 TS 08

8006

P/O 613425

C-85872

Jet Propulsion Lab
California Institute of Tech.
4800 Oak Grpve Drive
Pasadena, CA 91103

CERTIFICATE OF TEST
NOTICE OF SHIPMENT

WILL CALL

6AL/4V Titanium to MIL-T 9046 F Type 3 Comp C Ann.

	INVOICE NUMBER	DATE
	564 224	May 29, 1974

VIA	GROSS	TARE	NET
WILLCALL			2 1/2 lbs.

	DESCRIPTION	NO. PCS.	SQUARE FEET	BILLING WT. PER PIECE OR SQ. FT.	WEIGHT			BAL. DUE
1	.032 x RW x RL Heat Number K 8800 Test Number L 8283	2	2		2 1/2			C

CERTIFIED CHEMICAL ANALYSIS AND MECHANICAL PROPERTIES ON REVERSE SIDE

TITANIUM SUPPLY

CERTIFICATE OF TEST

CHEMICAL ANALYSIS

HEAT NO.	C	FE	N	AL	VA	CR	MO	H	ZR	SN	MN	O	
K 8800	.026	.09	.012	6.0	4.1			.011				.10	

MECHANICAL PROPERTIES

HEAT NO.	TEST NO.	SIZE OR GAUGE	.2% YIELD STRENGTH	TENSILE STRENGTH	ELONG.	R. A. %	HARDNESS	BEND TEST
		.032	KSI	KSI				R/T @ 20X
K 8800	L 8283	L	147	157	11			4.0
		T	138	152	10			4.0
		L	150	161	11			4.0
		T	130	149	10			4.0

MFG: TIMET

SUBSCRIBED AND SWORN TO BEFORE ME

...S DATE May 29, 1974

RESULTS AS ABOVE CERTIFIED
TITANIUM SUPPLY, GARDENA, CALIFORNIA

FUTURA TITANIUM CORPORATION RA691

SOLD TO:	SHIP TO:
Jet Propulsion Lab 4800 Oak Grove Drive Pasadena, CA 91103	Ref P/o 613425 C-85872

ITEM	CUSTOMER ORDER NO.	OUR ORDER NO.	DATE SHIPPED	DESCRIPTION	SIZE
1	GT 620091	5-15718	~~XXXXXXXX~~ 6-3-74	1pc.032 x 14 x 37 Titanium Sheet 6AL-4V STA	1pc-.032 x 14 x 37 (3.6#)

CHEMICAL ANALYSIS

HEAT #	C	FE	N_2	AL	VA	CR	MO	H_2	O_2	MN	TI	SN	NI	ELEMENTS
G-4764	.023	.06	.013	6.10	4.00			.006	.08		Bal			

PHYSICAL PROPERTIES

HEAT #	YIELD STRENGTH PSI	TENSILE STRENGTH PSI	ELONG % IN 2"	RED. OF AREA %	HARDNESS	REMARKS	TEST #
G-4764	151,200	164,000	14.0	37.5	39.5	Mfg: Timet Bend: OK	

SWORN TO AND SUBSCRIBED BEFORE ME

THIS 3 DAY OF ~~MAY~~ June 1974

NOTARY PUBLIC

CERTIFICATE OF TEST

THE TEST RESULTS SHOWN IN THIS REPORT ARE CORRECT TO THE BEST OF OUR KNOWLEDGE AND BELIEF.

FUTURA TITANIUM CORPORATION

CERTIFIED BY: ______________________ REPRESENTATIVE

FTSC—4 NCR—TR—2500—7/73

PACIFIC STEEL TREATING CO. PST

A 20733

6829 FARMDALE AVE. • NORTH HOLLYWOOD, CALIF. 91605
983-1450 877-3346

WT.

CUSTOMER: JET PROPULSION LABORATORY | PROC. ENG. | DATE: 6-7-4 | P.O. AT 613434

JOB DESCRIPTION

SHIPPER: L-85777

20 STRIPS .032 X 1½" X 6"

CUST. JOB NO. 778-RA691-1-2320

CONTAINERS REF. P/O 613425 C-85872

A/C PRIME

MATERIAL	CONDITION	SURFACE FINISH	MAX SECTION THICKNESS
6AL-4V TI	AGED	CAST, WROUGHT ☐ ROUGH MACH ☐ FINISH MACH ☒	.032

HARDNESS REQUIREMENTS	ROCKWELL	BRINELL	CORE	100% ☐	PARTIAL ☐	SPECIMEN ☐
TENSILE REQUIREMENTS	U/T KSI	YIELD KSI	ELONG %	R.A. %	NO. COUPONS TO PULL	

NO.	OPERATION	SPECIFICATION	MEDIUM	TEMP.	REQ'D SOAK TIME	FURN. NO.	DATE	TIME IN	TIME AT TEMP	TIME OUT	STAMP
				TITANIUM				A.M. P.M.	A.M. P.M.	A.M. P.M.	
				NO VAPOR DEGR. NO HYDROGEN				A.M. P.M.	A.M. P.M.	A.M. P.M.	
				NO NITROGEN				A.M. P.M.	A.M. P.M.	A.M. P.M.	
1	VAC PURGE RETORT							A.M. P.M.	A.M. P.M.	A.M. P.M.	PST HT 40
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
2	ANNEAL	MIL-H 81200A	AR	1425	30 MIN	43	6/8	12^10 P.M.	12^20 P.M.	12^50 P.M.	PST HT 40
3	RETORT COOL TO R/T							A.M. P.M.	A.M. P.M.	A.M. P.M.	PST HT 40
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
				TITANIUM				A.M. P.M.	A.M. P.M.	A.M. P.M.	
				NO VAPOR DEGR. NO HYDROGEN				A.M. P.M.	A.M. P.M.	A.M. P.M.	
				NO NITROGEN				A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	

3 TESTS

ACTUAL HARDNESS	ROCKWELL	BRINELL	CORE

D. Galloway
AUTHORIZED SIGNATURE

TITLE: appr. QC 2191

PACIFIC STEEL TREATING CO. PST

HEAT LOAD & SHIPPER CERTIFICATION **A43429**

6820 FARMDALE AVE. • NORTH HOLLYWOOD, CALIF. 91605
983-1450 877-3346

EXACT SOAK OPER.
OK TO EXTEND OPER.

OWNER	PROC. ENG.	DATE	P.O.
J.P.L.		8-11-75	GT 634235

JOB DESCRIPTION: 4 Test Specimens ✱

SHIPPER:
CUST. JOB NO.: 778-RT211-1-7320
CONTAINERS: 1 Paper Bag
A/C PRIME:

MATERIAL	CONDITION	SURFACE FINISH	SECTION THICKNESS
6AL-4V	A	CAST, WROUGHT ☐ ROUGH MACH ☐ FINISH MACH ☒	MAX. .050 MIN.

HARDNESS REQUIREMENTS — ROCKWELL: — BRINELL: — CORE: — 100% ☐ PARTIAL ☐ SPECIMEN ☐

TENSILE REQUIREMENTS — U/T: — KSI YIELD: — KSI ELONG: — % R.A.: — % NO. COUPONS TO PULL: —

NO.	OPERATION	SPECIFICATION	MEDIUM	TEMP.	REQ'D SOAK TIME	FURN. NO.	DATE	TIME IN	TIME AT TEMP	TIME OUT	STAMP
1	~~Deg~~ Clean	~~Vapor~~	Acetone Wipe					A.M. P.M.	A.M. P.M.	A.M. P.M.	
2	Age	mil # 81200A	Ag	1000	3 hr	22	8/11/75	10:00 A.M.	11:00 A.M.	2:00 A.M.	PST
	Retort cool Ar										PST

ORIGINAL PAGE IS OF POOR QUALITY

no test

TITANIUM
NO VAPOR DEGR.
NO HYDROGEN
NO NITROGEN

✱ 8-14-75
These specimens were scrapped. They were aged in a Hydrogen atmosphere for 1 hour by mistake then with argon as shown above.
Parts No G.
L. TOTH

SHIP. INSP.
LAB TESTS

ACTUAL HARDNESS — ROCKWELL: BRINELL: CORE:

AUTHORIZED SIGNATURE
TITLE

PACIFIC STEEL TREATING CO. PST

CERTIFICATION A43429

6820 FARMDALE AVE. • NORTH HOLLYWOOD, CALIF. 91605
983-1450 877-3346

EXACT SOAK OPER. ✓

OK TO EXTEND OPER.

CUSTOMER	PROC. ENG.	DATE	P.O.
J P L	[illegible]	8-11-75	GT 634235

JOB DESCRIPTION: 4 Test Specimens

SHIPPER:

CUST. JOB NO. 775-RT211-1-7320

CONTAINERS: 1 Plastic Bag

A/C PRIME:

MATERIAL	CONDITION	SURFACE FINISH	SECTION THICKNESS
6AL-4V	A	CAST. WROUGHT ☐ ROUGH MACH ☐ FINISH MACH ☒	MAX. .050 MIN.

HARDNESS REQUIREMENTS	ROCKWELL —	BRINELL —	CORE —	100% ☐ PARTIAL ☐ SPECIMEN ☐	
TENSILE REQUIREMENTS	U/T — KSI	YIELD — KSI	ELONG — %	R.A. — %	NO. COUPONS TO PULL —

NO.	OPERATION	SPECIFICATION	MEDIUM	TEMP.	REQ'D SOAK TIME	FURN. NO.	DATE	TIME IN	TIME AT TEMP	TIME OUT	STAMP
1	~~Deg~~ Clean	~~Vapor~~	Acetone Wipe					A.M. P.M.	A.M. P.M.	A.M. P.M.	[stamp]
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
2	Age	mil # 81200A	Arg	1000	3 h	22	8/11/5	10:00 A.M. P.M.	11:00 A.M. P.M.	2:00 A.M. P.M.	PST HT
	Retort cool Arg							A.M. P.M.	A.M. P.M.	A.M. P.M.	PST HT
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
	no test							A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	
								A.M. P.M.	A.M. P.M.	A.M. P.M.	

NO VAPOR DEGR.
NO HYDROGEN
NO NITROGEN

ORIGINAL PAGE IS OF POOR QUALITY

SHIP. INSP.	LAB TESTS		
ACTUAL HARDNESS	ROCKWELL	BRINELL	CORE

[signature] AUTHORIZED SIGNATURE

TITLE

CONSIGNEE/CC ACTOR

SHIP TO
PACIFIC STEEL TREATING
6829 FARMDALE AVE.
HOLLYWOOD, CALIFORNIA
ATTN:

SHIPPING – RECEIVING MEMORANDUM

JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

DATE	8-11-75
NO.	D-11522
PAGE	1 OF 1
PURCHASE ORDER/CONTRACT NO.	GT-634235
JOB NO.	778-RT211-1-7320
REQUESTED BY	STEADMAN/TOTH
DATE REC'D. AT DESTINATION	8-11-75
SHIPPED VIA	H/C
	[X] PREPAID [ ] COLLECT
MATERIAL LOCATION	241
SECURITY CLASSIFICATION	U

ITEM NO.	QUANTITY	DESCRIPTION	UNIT VALUE	PROPERTY ID NO.	RETURNABLE ITEM	ACCEPT	REJECT	BALANCE	ACCEPT	REJECT	BALANCE
1	4	TEST SPECIMENS TI-6AL-4V	$10.00	NONE	X						
		8-14-75									
		Parts scrapped by L. TOTH									
		NEED DATE 8-14-75									

(FOR JPL RECEIVING–SHIPPING USE ONLY)

INTERNAL DELIVERY: L. TOTH — BLDG. 125 — ROOM 224

EXPEDITOR: L. STEADMAN — BLDG. 171 — ROOM 235

PROPERTY OWNERSHIP: JPL/NASA

SHIPMENT AUTHORIZED BY: X L. STEADMAN

PACKED BY: JF

MANIFEST NO.

RECEIVED BY

DATE

INSTRUCTIONS
HEAT ~~XXX~~ TREAT SPECIMENS TO FULL HEAT TREATED CONDITIONS PER MIL-SPEC
CERTS REQUIRED. MIN DISTORTION. NOTIFY R. DUVALL, 354-3501 OF COST
BEFORE PROCEEDING.

RECEIPT NO.1		RECEIPT NO.2	
DATE REC'D.	PACK SLIP NO.	DATE REC'D.	PACK SLIP NO.
RECEIVED BY		RECEIVED BY	
INSPECTION NO.	DATE	INSPECTION NO.	DATE
SHIPPER NO.		SHIPPER NO.	

ORIGINATOR

JPL 0351 R 4/72

JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

INSPECTION REPORT

I. R. NO. A 1914

SHEET 1 OF 1

MO.	DAY	YR
7	17	7

PROGRAM	REF. DESIG.	S/N	NOMENCLATURE	P.O.	REQ. NO.
306	UK	NA	DELBENE	NA	NA

	SUB ASSY	ACCT. CODE NO.	REG. NO.	CONTRACT NO.
NA	NA 301	1210[illegible]	C[illegible]	NA

OPERATION AFFECTED	QTY. REC.		CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER	SUPPLIER
[illegible] FINISH	19	REQ.		Yes						Yes	JPL
		FURN.		NO						NO	

DWG/PART NO.	REV.	CONFIG.	WHERE INSP. PERFORMED	MRB NO.
10054227-1	B	NA	SOURCE JPL SAF ETR	

COG. ENG.	EXT.	LEVEL OF INSP.	TYPE HARDWARE	TRAVELER NO.
TOTH	7225	SHIP REC. IN-PROC. TEST FINAL	FLT. PROTO TA SE	NA

STATUS	SPEC. NO.	TYPE	LOT NO.	INSP. LEVEL CHK.
NA	NA	NA	NA	100%

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1		.005/IN FLATNESS CK'S .005/.009	UAI	use as is [illegible]	
2		.030 ± .001 DIM. CK'S .032/.034	UAI	use as is [illegible]	
3		NO MAT'L CERTS WITH PARTS	UAI	use as is [illegible]	
		NOTE: PENDING IDENT & PAS-SIVATION PER DWG.			

INSPECTED BY: W. Templin

SIGN, STAMP, DATE: W. Templin 7/18/75

ITEM	SUPPLEMENTARY INFO./CORRECTIVE ACTION
	All use as-is Rodell[illegible]

QTY. ACCP'T.	QTY. REJ.	PARTS REC. BY
19	0	Chester Wilson

COG. ENG.	DATE	Q. A. ENG.	DATE
L. Toth [illegible]			8-15-[illegible]

ACCOMPANY HARDWARE

JPL

JET PROPULSION LABORATORY
California Institute of Technology
Pasadena, Calif. 91103

INSPECTION REPORT

I. R. NO. A 3418

SHEET 1 OF 1

MO.	DAY	YR
9	[illegible]	[illegible]

PROJ. DESIG.	S/N	NOMENCLATURE	P.O.	REQ. NO.
[illegible] N/A	N/A	Metallic Specimen	N/A	N/A

	SUB ASSY	ACCT. CODE NO.	REG. NO.	CONTRACT NO.
R&D	[illegible]	3[illegible]-12103-0-3[illegible]	[illegible]	N/A

OPERATION AFFECTED	QTY. REC.
Dim. Insp.	11

DWG	REV.	CONFIG.
1[illegible]	B	N/A

WHERE INSP. PERFORMED: SOUTH / JPL / ETR

MRB NO.

	CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER
REQ.		N/A						
FURN.		N/A						

SUPPLIER: JPL

COG. ENG.	EXT.	LEVEL OF INSP.	TYPE HARDWARE	TRAVELER NO.
[illegible]	322[illegible]	SHIP / REC. / IN-PROC. / TEST / FINAL	FLT. / PROTO / TA / SE	N/A

STATUS	SPEC. NO.	TYPE	LOT NO.	INSP. LEVEL CHK.
NA	N/A	N/A	N/A	100%

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1		.030±.001 Thickness is up to .032 to .033 max.		[illegible]	
2		Flat within .005/in is OK one specimen (#3)		[illegible]	
3		No material certification		[illegible]	
4		No Passivation certification		[illegible]	
		Note: (1) Assigned test No's pending (2) See actual dims recorded on each envelope.		F. Toth 9-4-75	

INSPECTED BY: Joe [illegible] JPL 115

SIGN, STAMP, DATE

ITEM	SUPPLEMENTARY INFO./CORRECTIVE ACTION
	Note: These parts are [illegible] met [illegible] [illegible]
	F. Toth 9-4-75

QTY. ACCP'T.	QTY. REJ.	PARTS REC. BY
11		

COG. ENG.	DATE	Q. A. ENG.	DATE
F. Toth	9-4-75		

ACCOMPANY HARDWARE

JPL 1453 R 2/75

jpl → **MECHANICAL FABRICATION INSPECTION REQUEST**

REQ. NO.				
Q. NO.				
DATE 8-19-75	ENGINEER L. TOTH			P.O. NO.
REQUESTED BY L. TOTH	BLDG. 125	ROOM 224	EXT. 3215	I.R. NO. A 3413
DRAWING NO.	QUANTITY		SERIAL NO.	~~PROJECT NO.~~ 9-4-75
TITLE OR DESCRIPTION METALLIC SPECIMEN, DOUBLE DOGBONE				ACC. NO. 306-12103-0-3840

DETAILED INSTRUCTIONS, SKETCH, ETC.

Inspect parts per attached drawing dated 8-19-75 and as follows:

1. Ten (10) parts
   Compliance with 16√ finish
2. Six (6) parts
   Compliance with 64√ finish

Notes
1. Cross reference IR No. A 1914
2. Material is 6AL-4V Titanium, annealed condition. Notch on end of part is used to identify annealed condition (reference note 8 on dwg).
3. Determine typical surface finish of each part, and note value on each envelope

ORIGINAL PAGE IS OF POOR QUALITY

DELIVER TO	BLDG.	ROOM	DATE REQUIRED
P. WILLSON	125	224	8-29-75

Q.A. FILE

or sooner

SHIPPING — . ECEIVING MEMORANDUM

JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

CONSIGNEE/CO[illegible] CTOR

SHIP TO
Pacific Steel Treat
6829 Farmdale Ave.
Hollywood, Ca.

ATTN:

DATE: 6-6-74
NO.: C-85777
[illegible]GE: 1 OF 1
PURCHASE ORDER/CONTRACT NO.: GT-613434
JOB NO.: 778-RA691-1-7320 (6443)
REQUESTED BY: L. Potter
DATE REQ'D. AT DESTINATION: NS
SHIPPED VIA: H/C
[X] PREPAID [ ] COLLECT
MATERIAL LOCATION: 171-235
SECURITY CLASSIFICATION: U

ITEM NO.	QUANTITY	DESCRIPTION	UNIT VALUE	PROPERTY ID NO.	RETURNABLE ITEM	ACCEPT	REJECT	BALANCE	ACCEPT	REJECT	BALANCE
1	20 ea.	.032 x 9/16" x6" Titanium - 6AL-4V	$1.	NOne	X						
		Samples Cond.-Sol. H.T. & Aged									
2	1 Lot	Certify to MIL-H-81200	NCV	None	X						
		K. [illegible]	6-7								

The last six columns fall under the heading FOR JPL RECEIVING–SHIPPING USE ONLY.

INTERNAL DELIVERY: N/A
BLDG.:
ROOM:
EXPEDITOR: N/A
BLDG.:
ROOM:
PROPERTY OWNERSHIP: JPL/NASA
SHIPMENT AUTHORIZED BY: Duvall/Potter/BM
PACKED BY:
MANIFEST NO.:
RECEIVED BY:
DATE:

INSTRUCTIONS
Call R. Duvall (354-3501) on costs prior to processing . Anneal per spec.

RECEIPT NO.1		RECEIPT NO.2	
DATE REC'D.	PACK SLIP NO.	DATE REC'D.	PACK SLIP NO.
RECEIVED BY		RECEIVED BY	
INSPECTION NO.	DATE	INSPECTION NO.	DATE
SHIPPER NO.		SHIPPER NO.	

ORIGINATOR

Joth 125/224

JPL 0351 R 4/72

jpl → **MECHANICAL FABRICATION INSPECTION REQUEST**

DATE 7-8-74	ENGINEER L. Potter / W. Lewis			REG. NO. RA691 (12)
				~~R. NO.~~ Shipper C-85872
				P.O. NO. GT-613425
REQUESTED BY L. Potter	BLDG. 171	ROOM 235	EXT. 7179	I.R. NO.
DRAWING NO. 10056227-B	QUANTITY 32 + 7 spares		SERIAL NO.	PROJECT NO. 302
TITLE OR DESCRIPTION Titanium Specimens				ACC. NO. 775-RA-691-1-2320
= Supplier MAGNAFLUX TEST LAB				

DETAILED INSTRUCTIONS, SKETCH, ETC.

Inspect per furnished drawing except notes ~~[illegible]~~, 4, 5, 6 & 7 have not been accomplished.

COG. ENG.

SIGNATURE Louis Toth

DATE 7-18-74

JUL 8 RECD
MECHANICAL
QUALITY ASSURANCE

IR 256944
7-10-74

DELIVER TO	BLDG.	ROOM	DATE REQUIRED
W. Lewis	171	235	

QA FILE

JPL JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
4800 OAK GROVE DRIVE PASADENA CALIFORNIA

INSPECTION REPORT

I. R. NO. 28[illegible]

SHEET 1 OF 1

MO.	DAY	YR

PROGRAM	REF. DESIG.	S/N	NOMENCLATURE	P.O.	REQ. NO.
[illegible]	OK	NA	METALLIC SPECIMEN	[illegible]	[illegible]
R. & D.		SUB ASSY: NA	ACCT. CODE NO.: 502-R A 601-1-2330	REQ. NO.: NA	CONTRACT NO.: NA

OPERATION AFFECTED	QTY. REC.		CERTS	MAT'L	HEAT TREAT	STRESS R.	FLUR. PEN	X-RAY	ULTRA SON.	OTHER	SUPPLIER
DIM. INSP.	24	REQ.		YES	YES						MAGNETIC
DWG/PART NO.: 10058237-1-P	MRB NO.: NA	FURN.		YES	YES						TEST LAB

WHERE INSP. PERFORMED: SOURCE | JPL | SAF | AMR

MRB ITEM NO.:

COG. ENG.	EXT.	LEVEL OF INSP.	TYPE HARDWARE	TRAVELER NO.
LEWITZ	7174	REC. / IN-PROC. / TEST / FINAL	FLT. / PROTO / TA / OSE	NA

STATUS	SPEC. NO.	TYPE	LOT NO.	INSP. LEVEL CHK.
NA	NA	NA	NA	

ITEM	ZONE	DISCREPANCIES	DISP.	REMARKS	STAMP
1		.030 ± .0015 .0237 TO .029 ON 20 ANNEALED SPECIMENS.		Use as is LTOTH	
2.		.030 ± .0015 .0237 TO .027, 19 PCS, NOT ANNEALED.		Use as is LTOTH	
		NOTE: GEN. NOTES 4, 5, 6 & 7 PENDING.			

ORIGINAL PAGE IS OF POOR QUALITY

INSP. E. [illegible] JPL 280

DATE 7-17-74 [illegible] JPL 57

ITEM	CORRECTIVE ACTION
	R/AD item not for flight purpose -

QTY. ACCP'T.	QTY. REJ.	PARTS REC. BY

COG. ENG.	DATE	Q. A. ENG.	DATE
A. TOTH	7-18-74		

JPL 1453 (2-66)

ACCOMPANY HARDWARE

SHIPPING – RECEIVING MEMORANDUM

JET PROPULSION LABORATORY
California Institute of Technology
4800 Oak Grove Dr. / Pasadena, Calif. 91103

CONSIGNEE/CONTRACTOR:

SHIP TO:
M.C.I.
8100 E. Slauson Ave.
Montebello, Calif 90640

ATTN:

DATE: 2-3-76
NO.: D-21989
PAGE: 1 OF 1
PURCHASE ORDER/CONTRACT NO.: GT-649846
JOB NO.: 778-RS047-1-1950
REQUESTED BY: Steadman/Toth
DATE REC'D. AT DESTINATION: 2-3-76
SHIPPED VIA: H/C
☒ PREPAID ☐ COLLECT
MATERIAL LOCATION:
SECURITY CLASSIFICATION: U

ITEM NO.	QUANTITY	DESCRIPTION	UNIT VALUE	PROPERTY ID NO.	RETURNABLE ITEM	FOR JPL RECEIVING–SHIPPING USE ONLY: ACCEPT	REJECT	BALANCE	ACCEPT	REJECT	BALANCE
1	5	Ti-6A-4V Specimens DWG#10056227B	25.00	None	X						
2	1	DWG #10056227C	NCV	None	X						
		"NOTES"									
		1.Parts are finished machined									
		2.DO NOT EXPOSE to Halogenated									
		2.Solvents such as freon for cleaning									
		3.Notes 1 thur 7 not applicable to this order									
		Need Date: 2-6-76									

INTERNAL DELIVERY: P. Wilson — BLDG. 125 — ROOM 224
EXPEDITOR: Steadman — BLDG. 171 — ROOM 235
PROPERTY OWNERSHIP: JPL/NASA
SHIPMENT AUTHORIZED BY / PACKED BY: R.Duvall/L.Steadman/ar
MANIFEST NO.:
RECEIVED BY:
DATE:

RECEIPT NO.1 — DATE REC'D. / PACK SLIP NO. / RECEIVED BY / INSPECTION NO. / DATE / SHIPPER NO.
RECEIPT NO.2 — DATE REC'D. / PACK SLIP NO. / RECEIVED BY / INSPECTION NO. / DATE / SHIPPER NO.

INSTRUCTIONS:
Anneal Specimens in Vacuum or inert Atmosphere per Mil-T-009046G.
Notify R.Duvall, 354-3501, of costs before proceeding

ORIGINATOR

JPL 0351 R 4/72

APPENDIX C

FLUORINE IMMERSION STORAGE PROCEDURE

TRW
SYSTEMS GROUP
ONE SPACE PARK • REDONDO BEACH, CALIFORNIA

CODE IDENT 11982

TITLE

STORAGE IN LIQUID NITROGEN OF SEALED CAPSULES CONTAINING TITANIUM SPECIMENS AND/OR LIQUID FLUORINE

DATE 8 NOVEMBER 1977 NO. D01755

SUPERSEDING: New

PREPARED BY: J R Denson
J. R. DENSON

APPROVAL SIGNATURES:

D Wever 11/14/77
D. M. WEVER, Manager DATE
Capistrano Chemical Facility

B B Milburn 11/18/77
B.B. MILBURN, Manager DATE
CTS Health and Safety

for R L McGath 11/21/77
F. R. GARBARINE, Manager DATE
Capistrano Test Site

DATE

DATE

DATE

SYSTEMS 1391 REV. 5-67

DOCUMENT NO. D01755

TRW
DEFENSE AND SPACE SYSTEMS GROUP

ONE SPACE PARK • REDONDO BEACH • CALIFORNIA, 90278

REVISION RECORD

REV	DATE	AUTHORIZATION	CHANGE DESCRIPTION	PAGES AFFECTED
New	11-8-77	See Title Page	New Procedure	All

1.0 SCOPE

1.1 This document describes the storage of canisters loaded with specimen capsules in a temperature conditioning flask that is filled two times a week with liquid nitrogen. There are a total of thirty (30) capsules; eight capsules contain about 10 ml of liquid fluorine and twenty-two capsules contain a titanium specimem and about 10 ml of liquid fluorine. It is intended that the titanium specimens and liquid fluorine remain in storage for a period up to five years.

2.0 CONDITIONS

2.1 Personnel

2.2.1 Members of the Technical Staff and Technicians, as required.

2.2 Safety

Personnel shall comply with the TRW-CTS Facility Safety requirements and the Safety Requirements for the Chemistry and Chemical Engineering Laboratory. When adding liquid nitrogen for the first five minutes to the CRYO-FLASK, personnel shall remain behind a barricade. Safety of the storage method was evaluated and documented in the Applicable Documents listed in Sections 2.3.3 and 2.3.4. Capsules opened in a dewar containing liquid nitrogen caused no problems, but permitted slow distillation of fluorine from the dewar over a 24-hour period.

2.3 Applicable Documents

2.3.1 Safety Requirements for the Chemistry and Chemical Engineering Laboratory.

2.3.2 D-0051 (FTD 13.1) Safety Requirements, CTS.

2.3.3 2316-6020-RU-00, "Compatibility Testing of Spacecraft Materials and Space-Storable Liquid Propellants", Final Report, February 1974

2.3.4 16681-6009-RO-00, "A Plan for a Material Compatibility Program for Oxygen Difluoride (OF_2) and Diborane (B_2H_6) Liquid Propellants", Final Report, April 1971

2.4 Test Equipment

2.4.1 Storage Bunker

2.4.2 CRYO-FLASK, Model A-1500, Liquid Nitrogen, Manufactured by Minnesota Valley Engineering Company

2.4.3 Liquid Nitrogen Supply Tank

2.4.4 Test Set-up (See Figure 1)

2.5 Calibration

All instruments used to obtain measurements for data acquisition shall be calibrated in accordance with TRW Quality Assurance Standard Practice and within current acceptable calibration periods.

3.0 OPERATION

3.1 Verify that the liquid nitrogen supply is connected to the CRYO-FLASK.

3.2 Verify that the level indicator on the liquid nitrogen supply tank indicates that the tank is at least 25% full.

3.3 Verify that the pressure gauge on the liquid nitrogen supply tanks indicates at least 10 psig pressure in the tank.

NOTE

Be certain that all personnel are behind a barricade (the wall of the storage bunker).

3.4 Open full the liquid delivery valve on the liquid supply tank.

3.5 Observe when the CRYO-FLASK is full of liquid nitrogen. This observation is made by noting when fog coming from the bunker increases greatly. At this time the liquid nitrogen overflowing from the CRYO-FLASK can be observed.

NOTE

Observation of the CRYO-FLASK is safe at all times, but as a precaution, it shall not be observed during the first five minutes of filling. The CRYO-FLASK in use retains 25% of the liquid nitrogen after standing for four weeks.

3.6 Close the valve on the liquid nitrogen supply tank when the CRYO-FLASK is filled with liquid nitrogen.

3.7 Record in a notebook the date and time the CRYO-FLASK is filled with liquid nitrogen.

PREPARED ____
CHECKED ____
MODEL ____

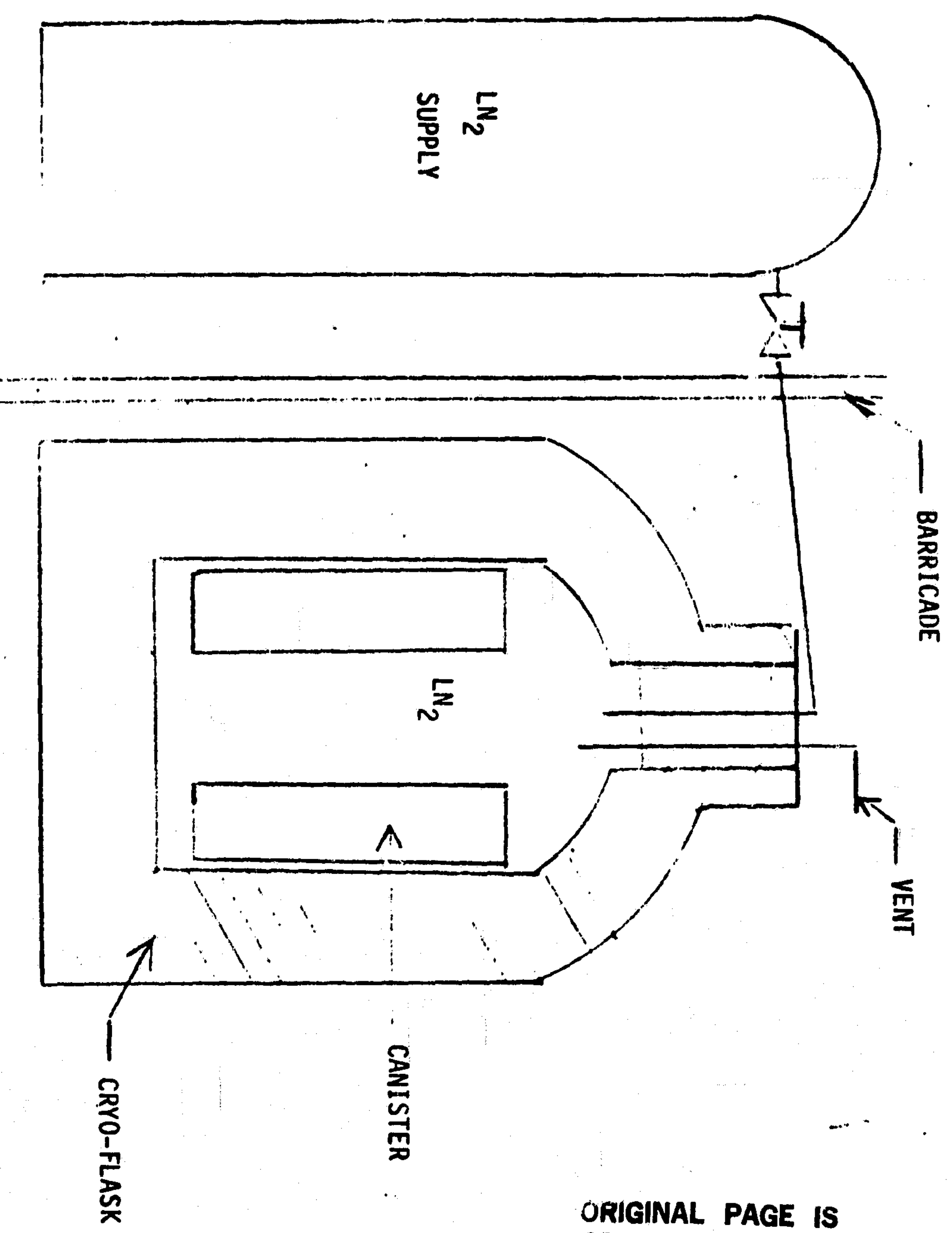


FIGURE 1 - CONDITIONING FLASK

APPENDIX D

PROCEDURE FOR TEST TERMINATION

TRW

DEFENSE AND SPACE SYSTEMS GROUP

ONE SPACE PARK • REDONDO BEACH, CALIFORNIA

FSCM NO. 11982

TITLE

REMOVAL OF FLUORINE AND ANALYSIS OF FLUORINE IN LONG TERM STORAGE TESTS OF TITANIUM SPECIMENS WITH LIQUID FLUORINE

DATE 18 Oct 1978

NO. YS-28T-15

SUPERSEDING: ____________

____

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DOCUMENT NO. YS-28T-15

TRW
DEFENSE AND SPACE SYSTEMS GROUP

ONE SPACE PARK • REDONDO BEACH • CALIFORNIA, 90278

REVISION RECORD

REV	DATE	AUTHORIZATION	CHANGE DESCRIPTION	PAGES AFFECTED
New	10-18-78	See Title Page	New Procedure	All

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SYSTEMS 1392 REV.1-77

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1.0 SCOPE

1.1 In July 1976 capsules manufactured from glass were sealed with liquid fluorine or with titanium specimens and liquid fluorine. Since sealing these capsules have been in storage at -196°C. This procedure describes the twice before performed safe removal of liquid fluorine from the long term stored capsules.

2.0 CONDITIONS

2.1 <u>Personnel</u>

Chemists and Technicians, as required. Personnel shall be trained in the normal operations performed in handling fluorine.

2.2 Personnel shall comply with TRW-CTS Facility Safety Requirements and with the Chemistry and Chemical Engineering Laboratory Safety Requirements. Personnel shall be thoroughly acquainted with the Fluorine and Liquid Nitrogen Sections of the Safety Requirements. for the Chemistry and Chemical Engineering Laboratory. A safety face shield, gloves and rubber apron shall be worn when performing all operations. While working in a hood with capsules containing fluorine, a safety shield shall be placed between the operator and capsules.

All operations described in this document were performed two times during the last five years.

2.3 <u>Applicable Documents</u>

2.3.1 Safety Requirements for the Chemistry and Chemical Engineering Laboratory.

2.3.2 Capistrano Test Site Manual, Part III, Safety Procedures and Part IV, Emergency Procedures.

2.3.3 Matheson Gas Data Book, section on fluorine.

3.0 REMOVAL OF LIQUID FLUORINE (LF_2) FROM CAPSULES AND ANALYSIS OF RESIDUE

3.1 Removal of LF_2 from Capsules

NOTE

In an emergency such as observing a broken capsule, the capsule shall be placed in a Dewar flask filled with LN_2. This Dewar flask shall then be placed in the test cell. Under these conditions, the LF_2 will boil off overnight with no problems except the slow release of F_2 as a gas.

NOTE

Operators shall wear face shield, laboratory coat, rubber apron, and asbestos gloves when handling cold capsules.

3.1.1 Move the liquid nitrogen (LN_2) filled Cryo-Flask to the oxidizer test cell.

3.1.2 Fill a ten-inch deep Dewar with LN_2.

3.1.3 Place the LN_2 filled Dewar next to the Cyro-Flask.

3.1.4 Lift by the attached handle the canister containing the capsule to be sampled to a level where one-half the canister is covered with LN_2.

NOTE

A canister is a stainless steel tube with a screen covered bottom and a long handle that retains 8 capsules while immersed in LN_2 contained in the Cyro-Flask.

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3.1.5 By the second operator, move the LN_2 filled Dewar to a position next to the Cryo-Flask neck.

NOTE

Two operators must work as a team when capsules containing LF_2 are moved.

3.16 Rapidly lift the canister from the Cryo-Flask.

3.1.7 Rapidly move the capsule being sampled from the canister and place in the LN_2 filled Dewar.

3.1.8 Immediately place the canister below the LN_2 level in the Cryo-Flask.

3.1.9 Move the LN_2 filled Dewar containing the capsule to the hood contain-in the Fluorine Handling Manifold and replace the chain across the entrance to the test cell.

NOTE

A safety shield shall be between the operator and the capsule while the capsule is in the hood.

3.1.10 While immersed in LN_2, manipulate the specimen capsule into position for connection to the opening device shown in Figure 1 which is connected to handling manifold shown in Figure 2.

NOTE

The handle to the opening device valve is extended so that it passes through a small hole in a safety shield placed in front of the opening device.

3.1.11 Connect the specimen capsule to the opening device using a Swagelock nut and Teflon ferrules.

3.1.12 Close or verify closed Valves 1, 2, 3, 5, 6, 8, 9, and 10.

3.1.13 Open or verify open Valves 4, 7, and 11.

3.1.14 Verify the Manifold Pump is on and that the Dewar flask and the LN_2 cooled traps are full.

3.1.15 Connect at Valve 1 a 10 cm stainless steel infrared cell fitted with AgCl windows.

3.1.16 Open Valves 1 and 2.

NOTE

Allow the tip of the specimen capsule to remain in vacuum for five minutes to remove any moisture on the surface of the capsule tip.

3.1.17 Close Valve 7 and observe the vacuum gauge for three minutes to determine there are no leaks.

3.1.18 Using the handle extension on the opening device, turn the handle 90° which will crush the tip off the specimen capsule.

3.1.19 Return the handle on the opening device to the original position.

3.1.20 Record the pressure noted in the manifold.

NOTE

The volume of the manifold with a capsule and infrared cell connected shall have been determined prior to this.

3.1.21 Verify that the vacuum pump in the test cell is on.

3.1.22 Open Valve 6 and vacuum distill the LF_2 from the specimen capsule.

NOTE

The LF_2 is passed through a soda lime and then a charcoal scrubber before entering the vacuum pump. A thermocouple is attached to the soda lime scrubber about 20% of the distance down the soda lime packing in the copper tank. If the thermocouple measures a temperature of 48°C, the distillation is stopped until the scrubber cools to 30°C.

3.1.23 When the distillation is complete as determined by the observation that no liquid remains, close Valve 6.

3.1.24 Remove the LN_2 filled Dewar from the sample capsule.

3.1.25 Record the pressure in the manifold when the sample capsule is free of frost and the capsule temperature is near room temperature as determined by touch.

3.1.26 Close Valve 1 and the valve on the infrared cell.

3.1.27 Open Valve 7 and evacuate the manifold for five minutes.

3.1.28 Remove the infrared cell and analyze its contents as indicated in Section 3.2.

3.1.29 Close Valve 7.

3.1.30 Bleed helium through Valve 8 until the pressure in the manifold is 760 torr.

3.1.31 Close Valves 8 and 2.

3.1.32 By means of a propane-oxygen torch seal the tip of the specimen capsule.

NOTE

Ship the specimen capsules to the Metallurgical Laboratory at Space Park for examination. The material being shippedis not hazardous, and does not require special handling techniques.

3.1.33 Remove the broken glass tip from the Capsule Opening Device.

3.2 Residue Analysis

3.2.1 Obtain standard calibration curves for each of the substances listed below at the absorption frequencies listed:

Substance	cm^{-1}
HF	3770
CO_2	2350
CF_4	1280
SiF_4	1050

3.2.2 Obtain an infrared spectrum between 4000 cm^{-1} and 750 cm^{-1} of the residue collected at -196°C and evaporated at room temperature (see 3.1.3).

3.2.3 Read concentration for each species present from Standard Curves.

NOTE

To determine the concentration of each species it will be necessary to calibrate the volume of the manifold and the infrared cell.

3.2.4 Perform calculation of species concentration

$$\text{mg of species} = \frac{\% \times V \times MW}{22.5 \times 100}$$

where:

% = concentration from standard curve in %

V = volume of manifold and infrared cell

MW = molecular weight of species

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LTR	DESCRIPTION	DATE	APPROVED

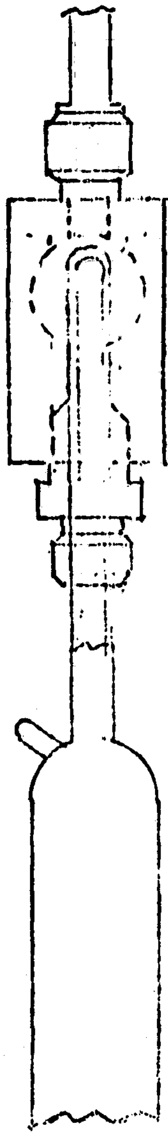

Figure 1
Capsule Opening Device

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LTR	DESCRIPTION	DATE	APPROVED

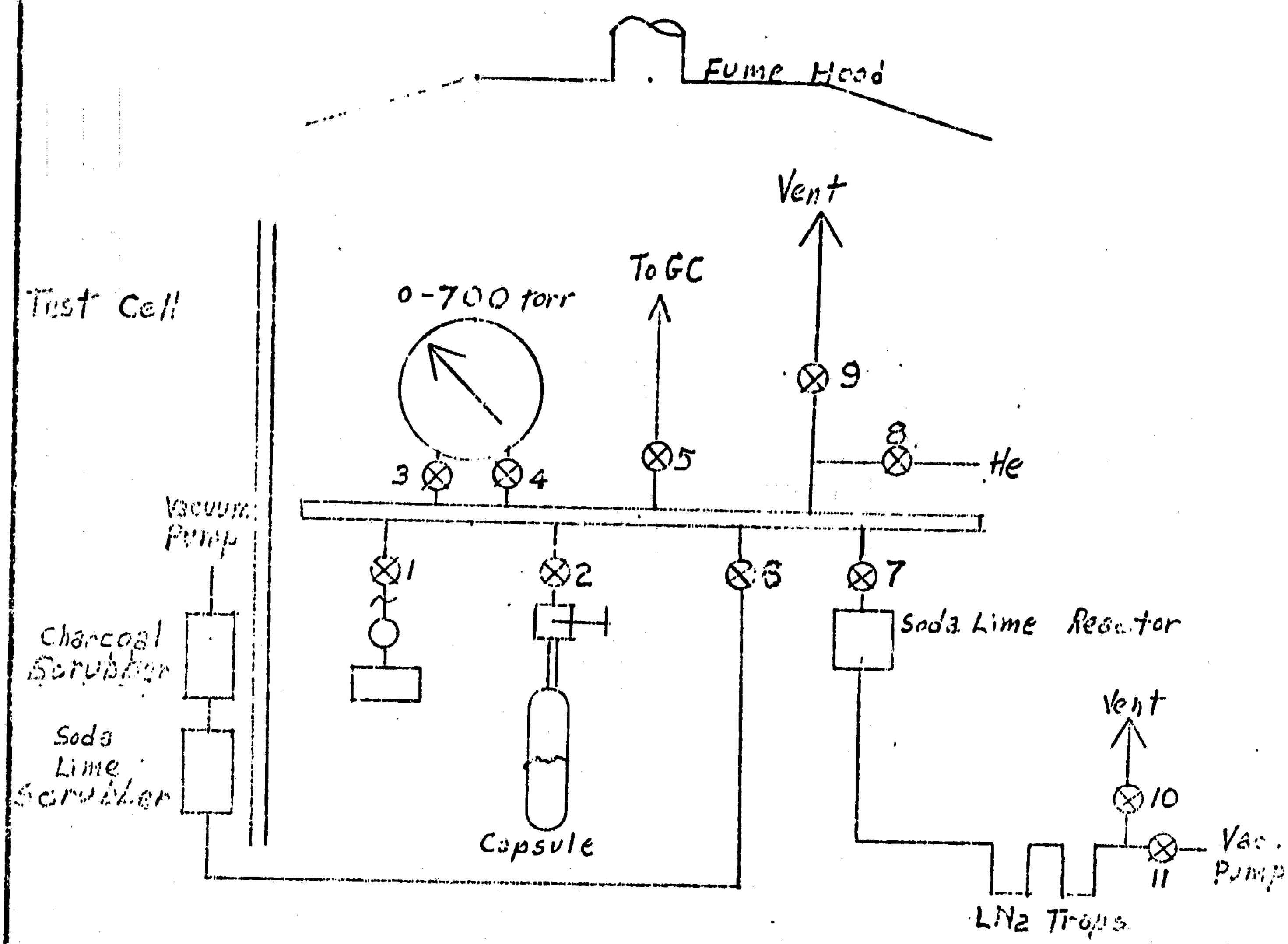

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Figure 2
Fluorine Handling Manifold

ENGINEERING SKETCH		TRW SYSTEMS GROUP ONE SPACE PARK • REDONDO BEACH, CALIFORNIA
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		SCALE | SHEET 1 OF

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APPENDIX E

APPEARANCE OF PRE-IMMERSION COUPONS

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Figures

Tables

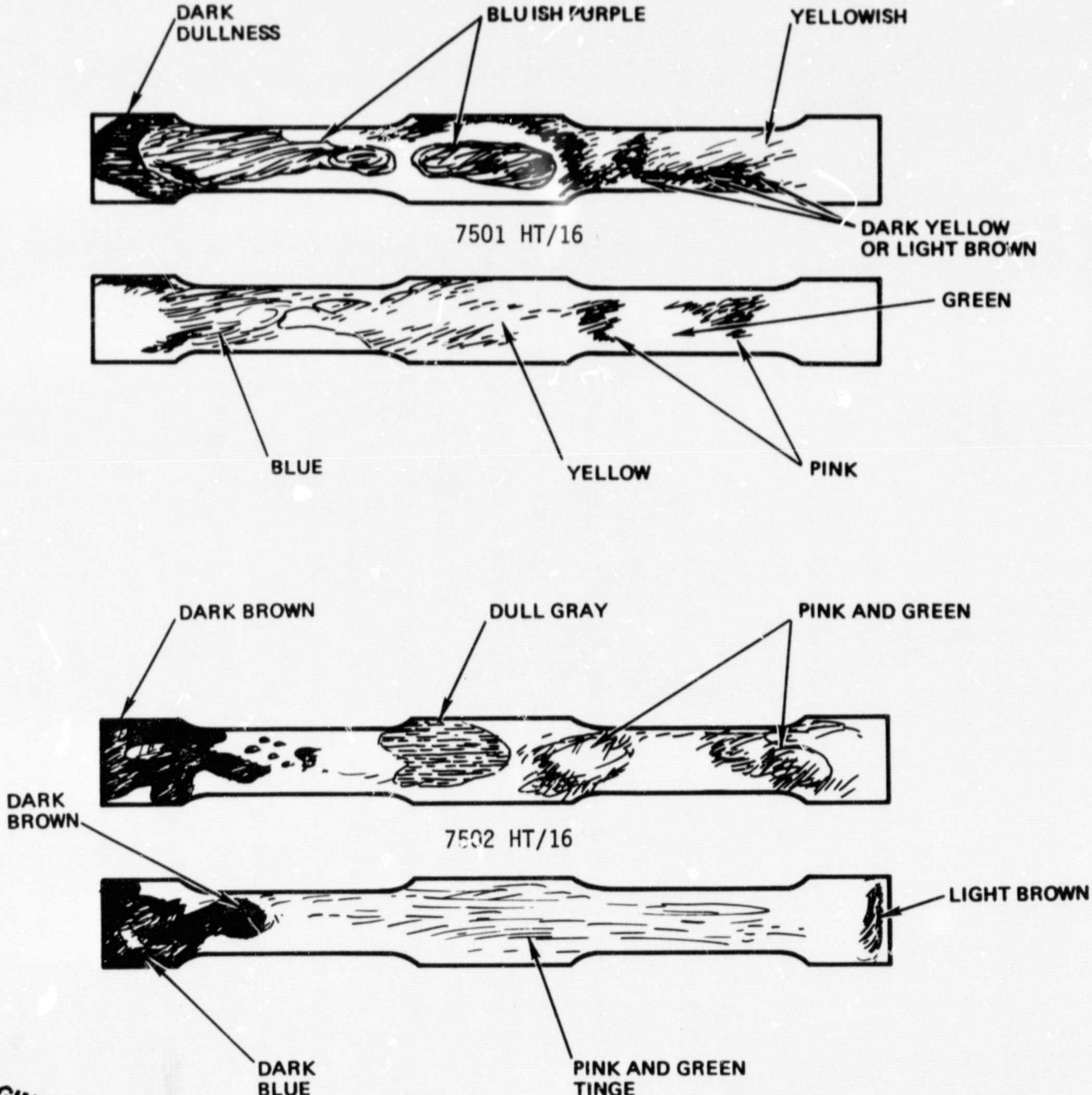


Figure E-1. Maps of Surfaces of Passivated Specimens

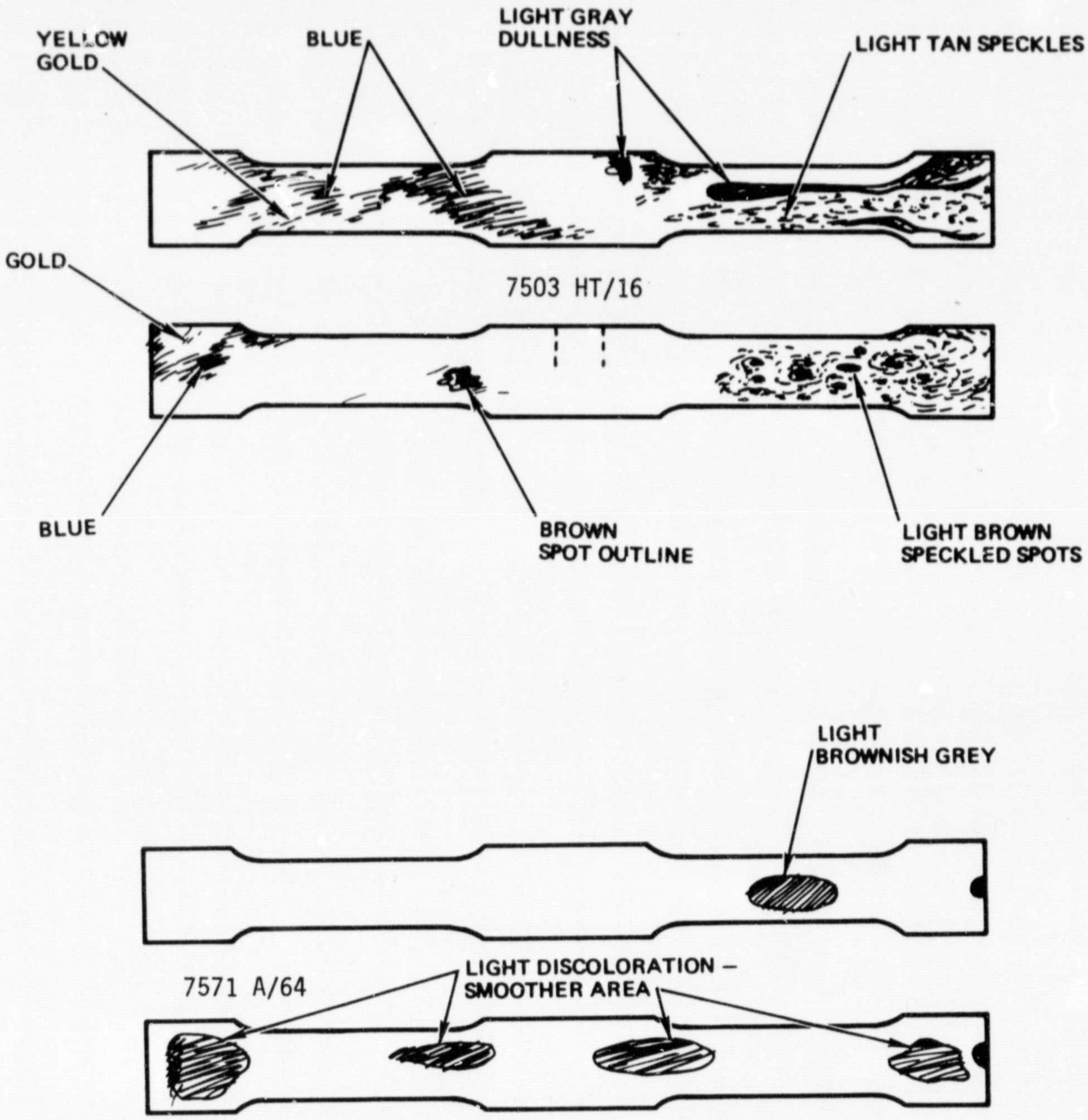


Figure E-1: Maps of Surfaces of Passivated Specimens (Continued)

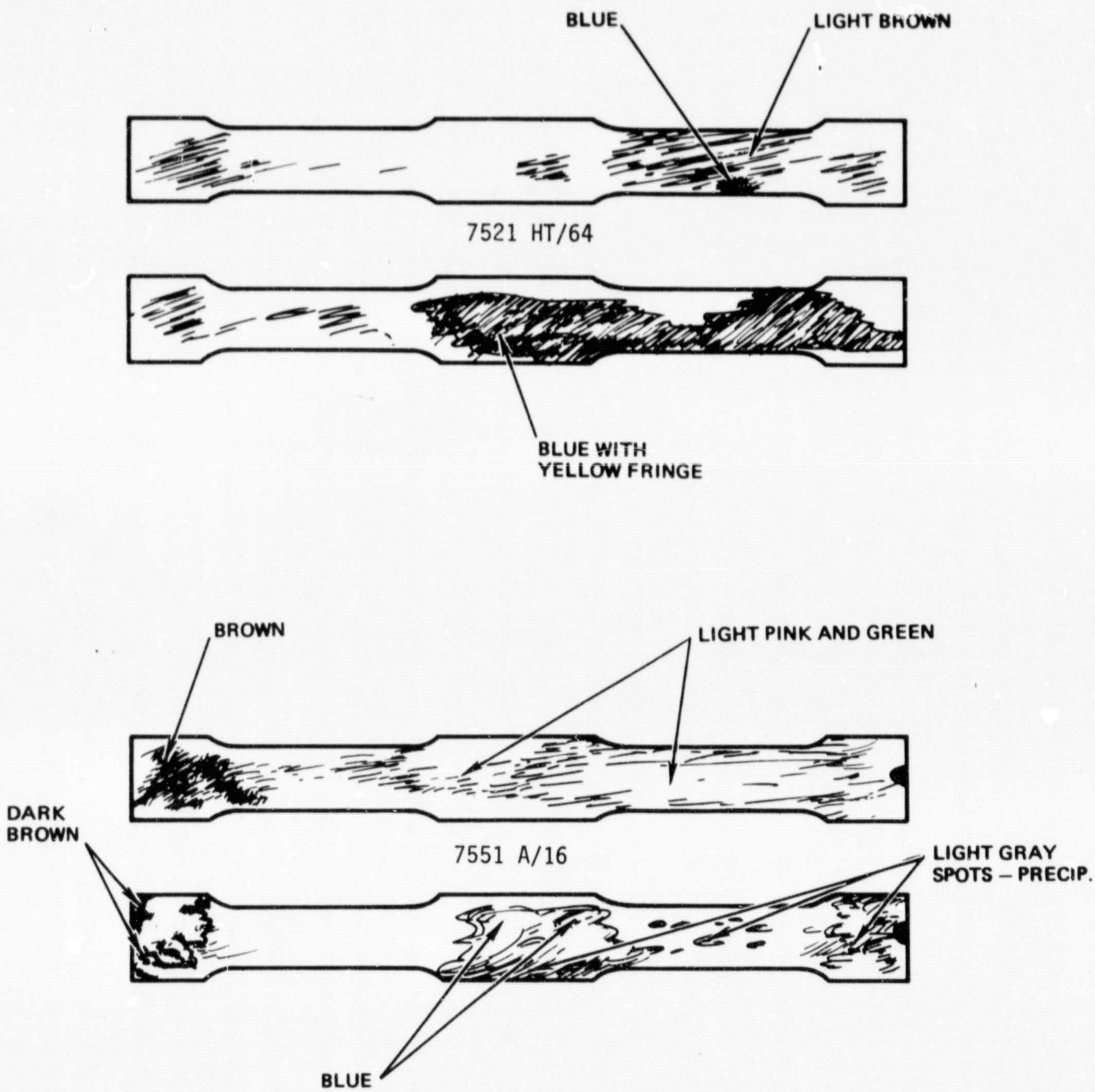
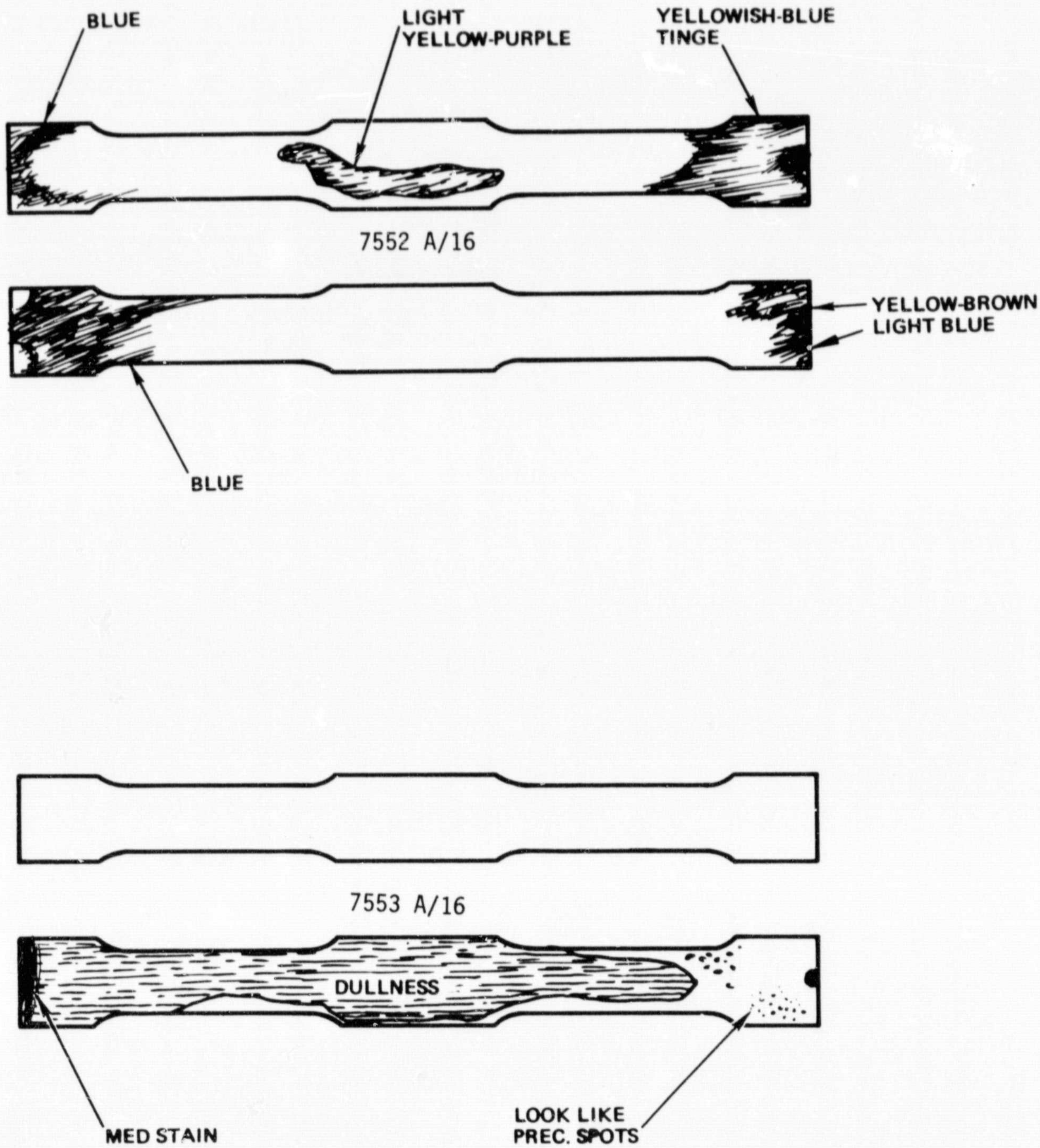


Figure E-1: Maps of Surfaces of Passivated Specimens (Continued)



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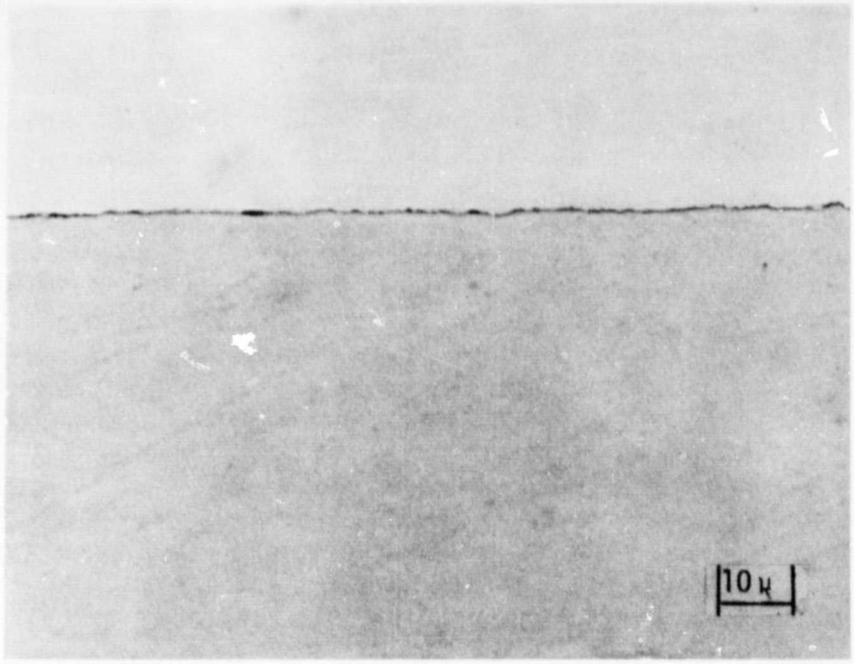

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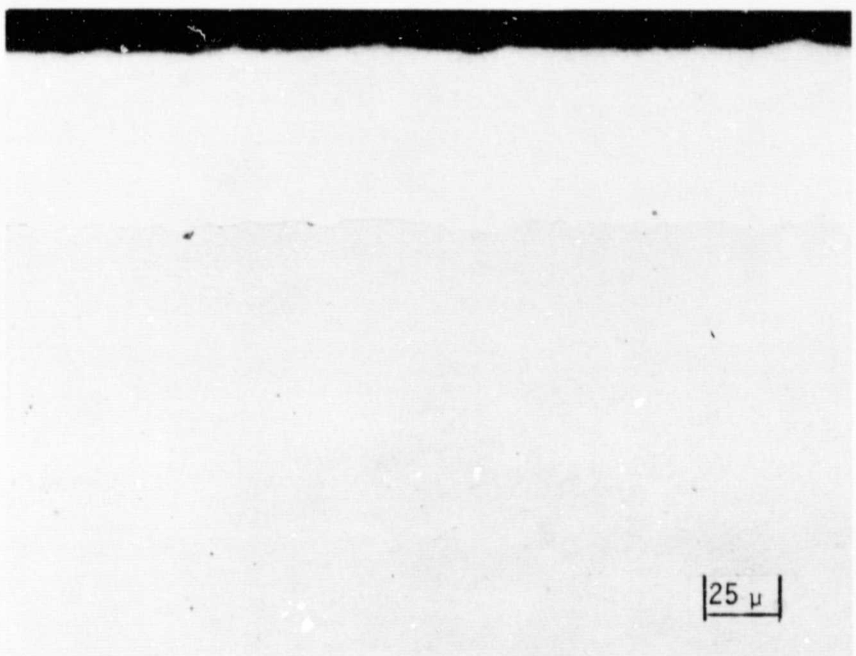
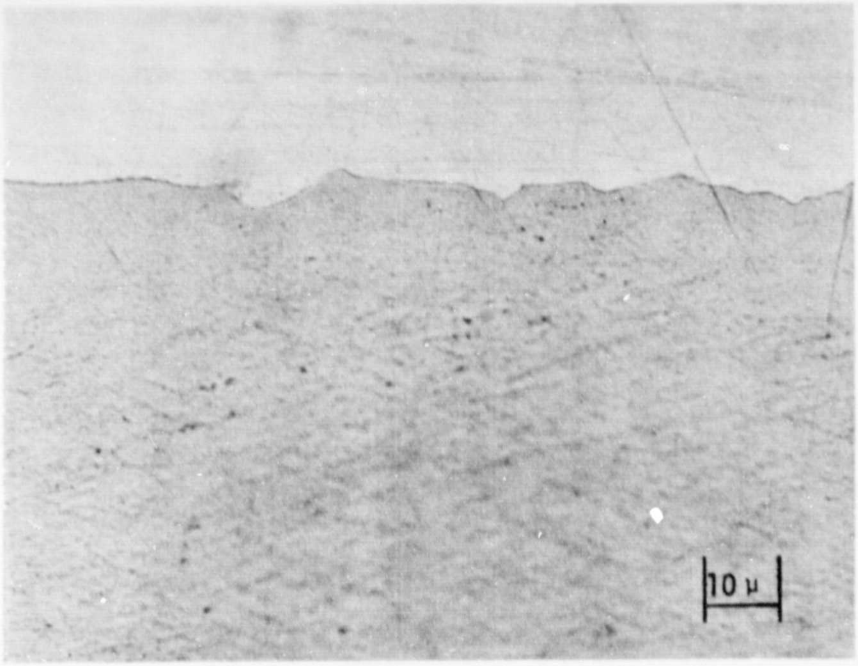
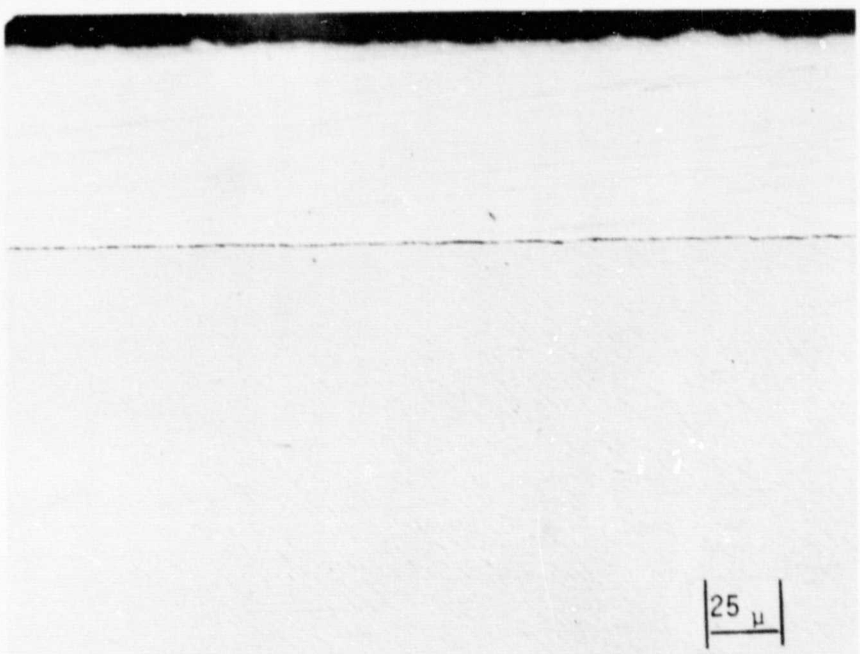


b) 7520 H/64 (∼400 X)

Figure E-2: Photomicrographs of As-Received Specimens

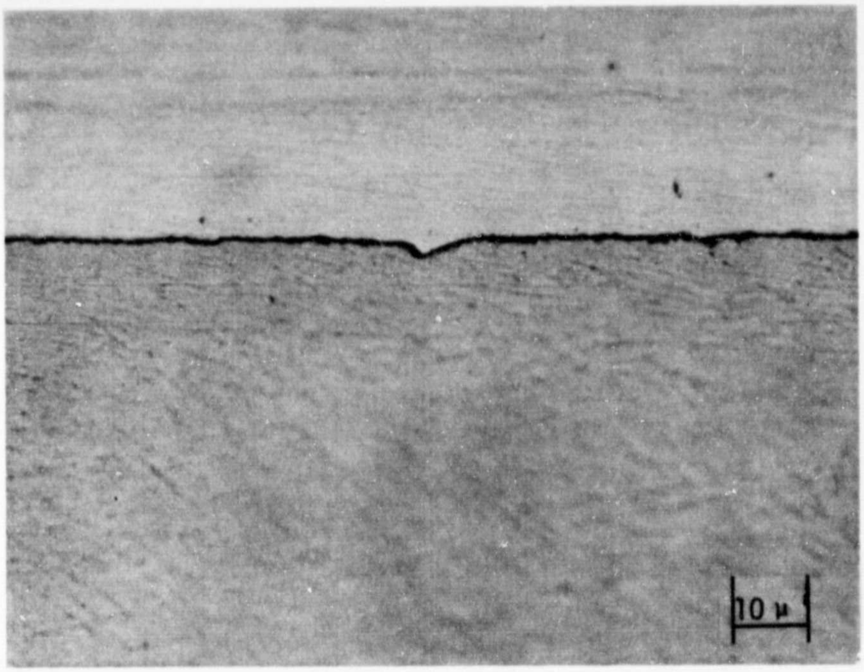


c) 7520 H/64 (~1000 X)

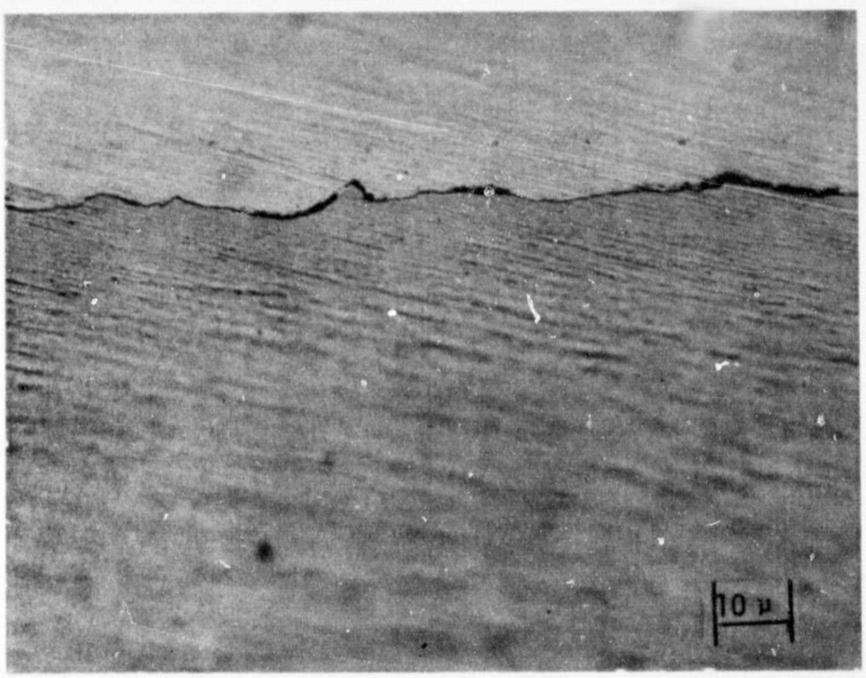


d) 7550 A/16 (~400 X)

Figure E-2: Photomicrographs of As-Received Specimens (Continued)

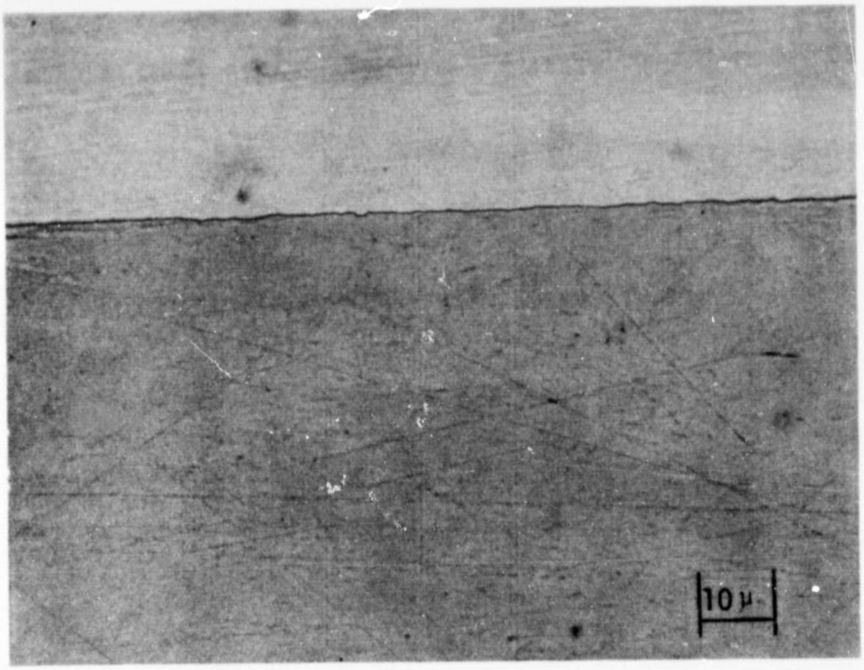


e) 7550 A/16 (~ 1000 X)



f) 7570 A/64 (~ 1000 X)

Figure E-2: Photomicrographs of As-Received Specimens (Continued)



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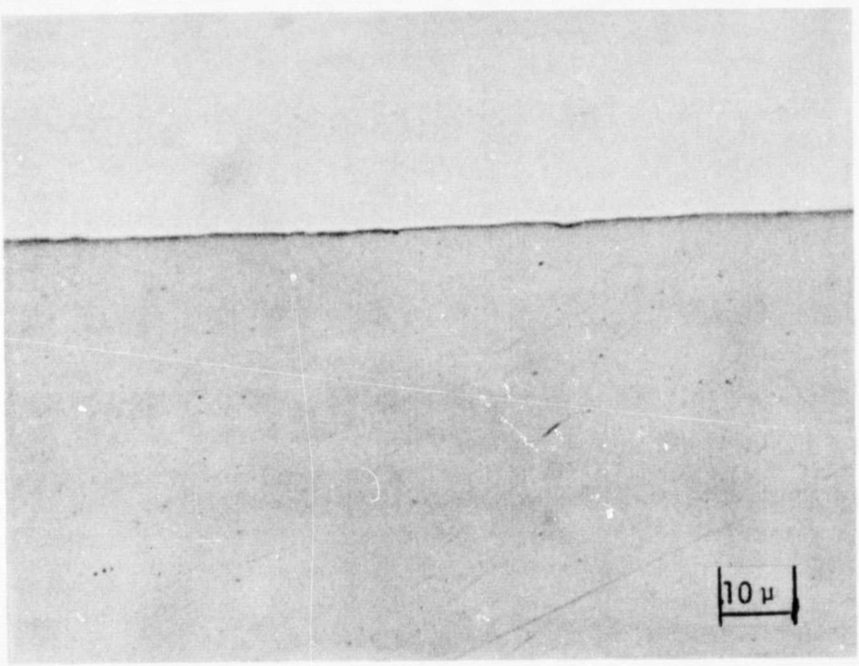
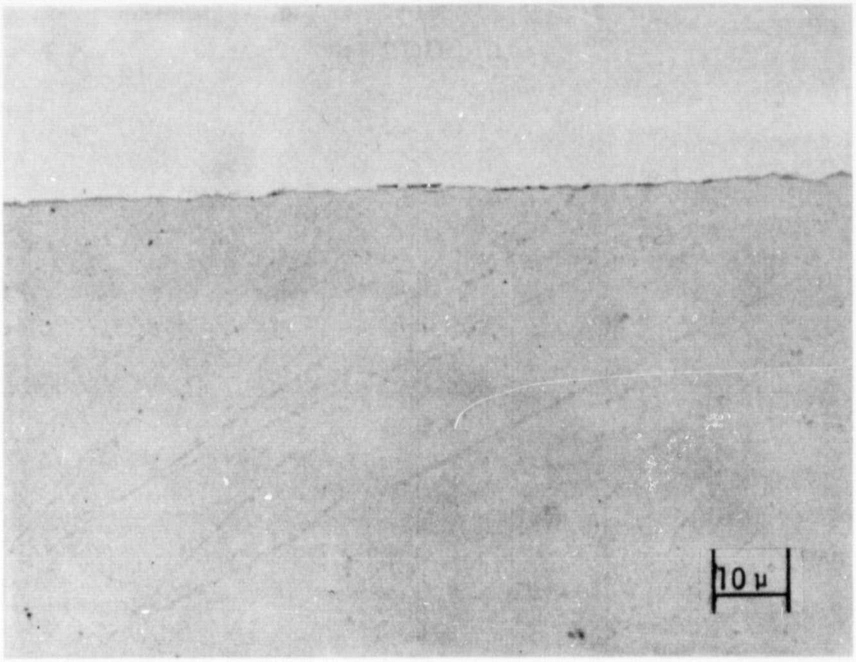
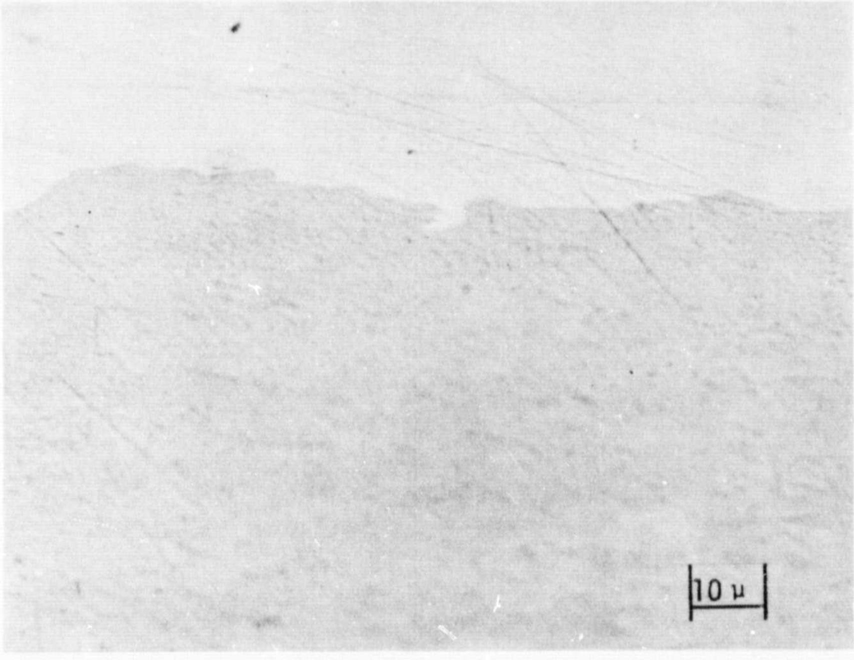


b) 7502 H/16 (~1000 X)

Figure E-3: Photomicrographs of Passivated Specimens



c) 7503 H/16 (∼1000 X)



d) 7521 H/64 (∼1000 X)

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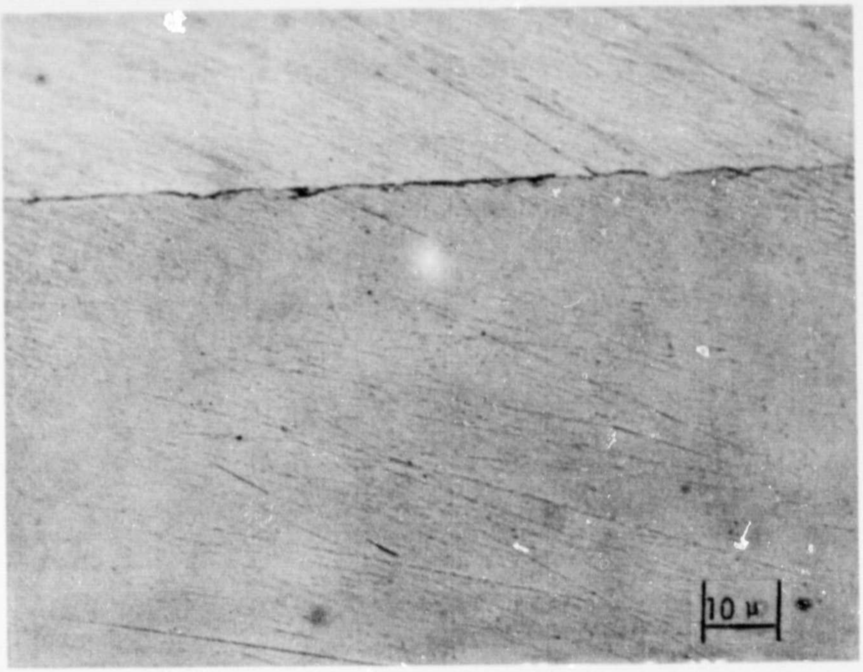
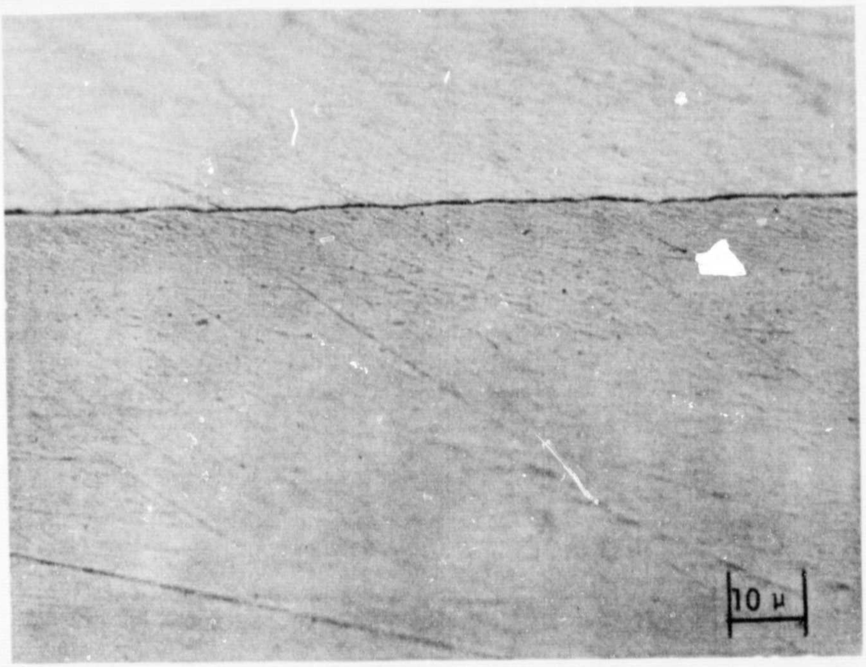


e) 7552 A/16 (∼ 1000 X)



f) 7553 A/16 (∼ 1000 X)

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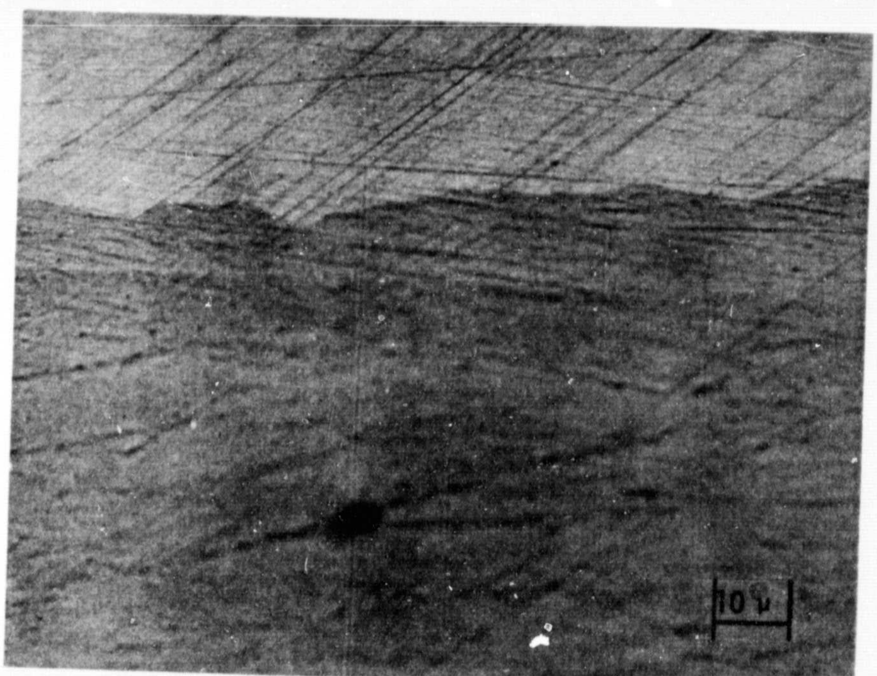


g) 7571 A/64 (~1000 X)

Figure E-3: Photomicrographs of Passivated Specimens (Continued)

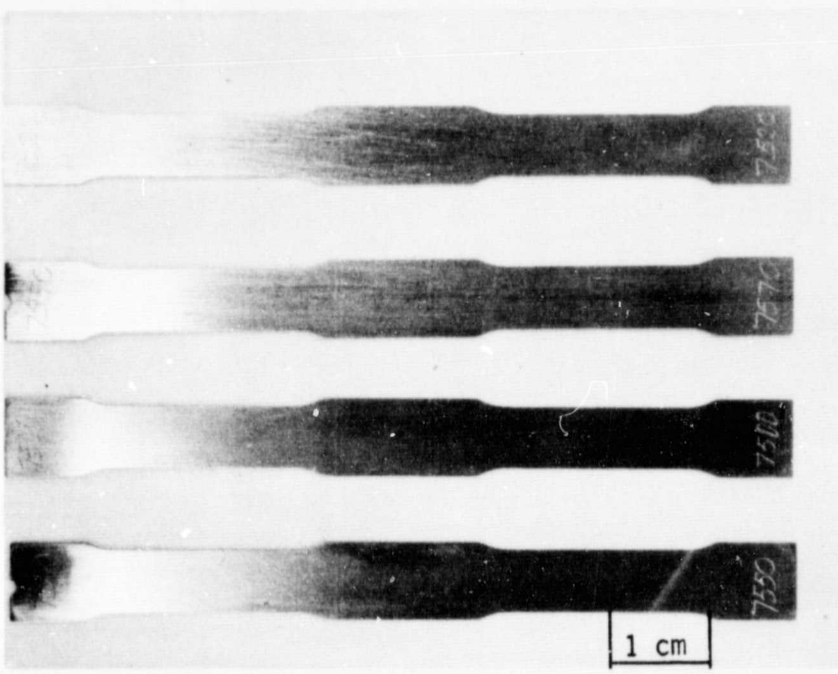
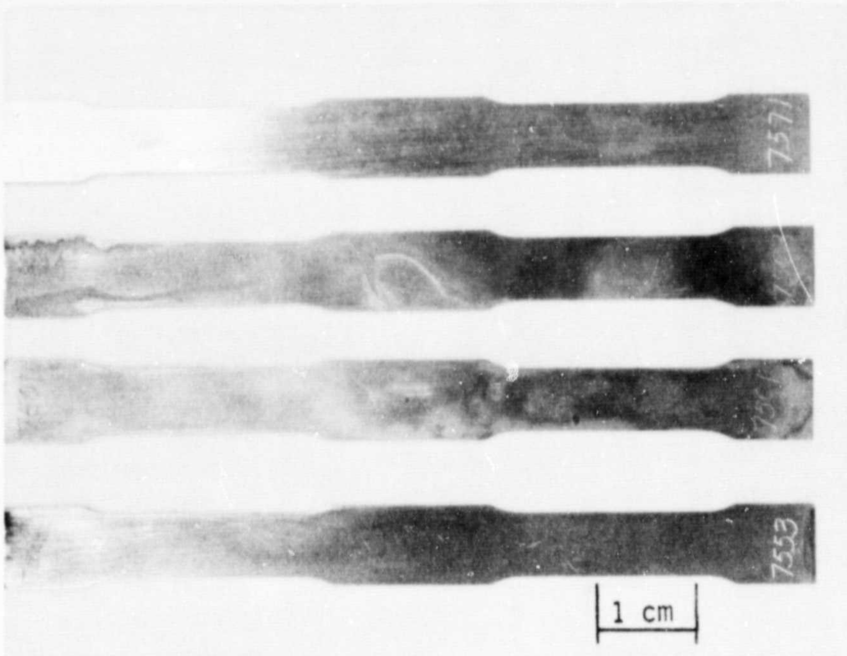
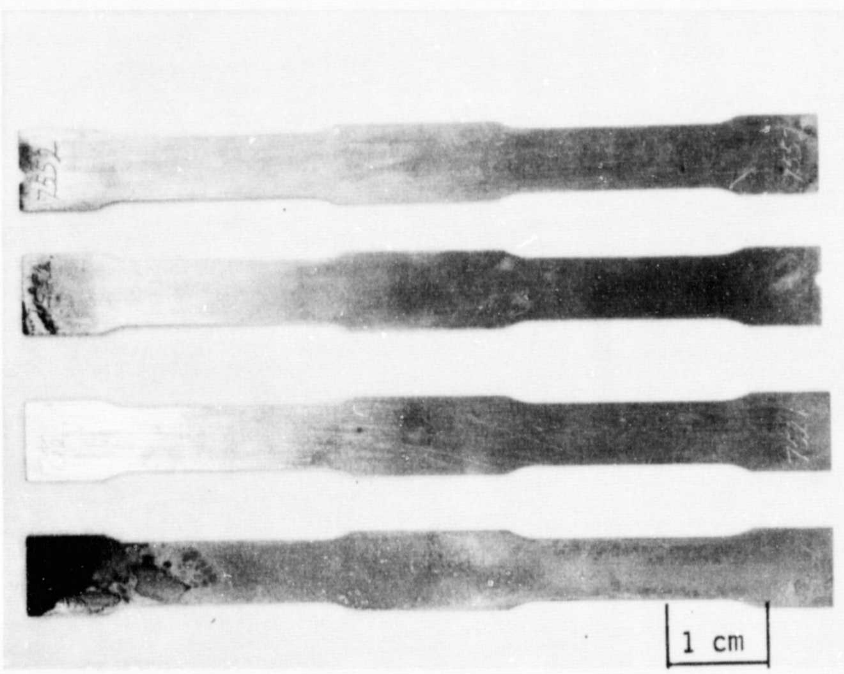


Figure E-4: Photographs of Unpassivated Specimens
Top to bottom: 7520, 7570, 7500, 7550.

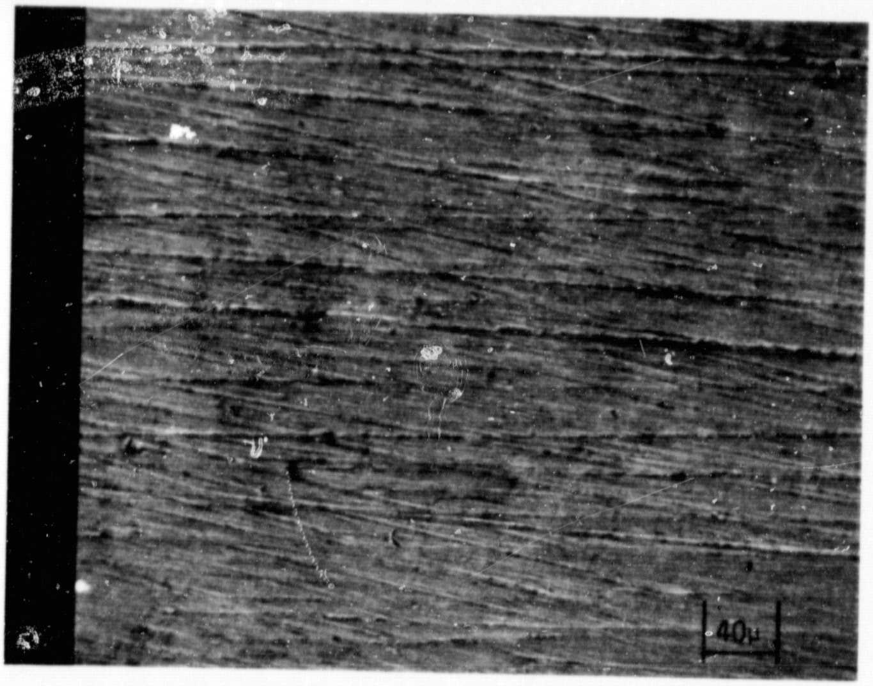


a) Top to bottom: 7571, 7503, 7501, 7553

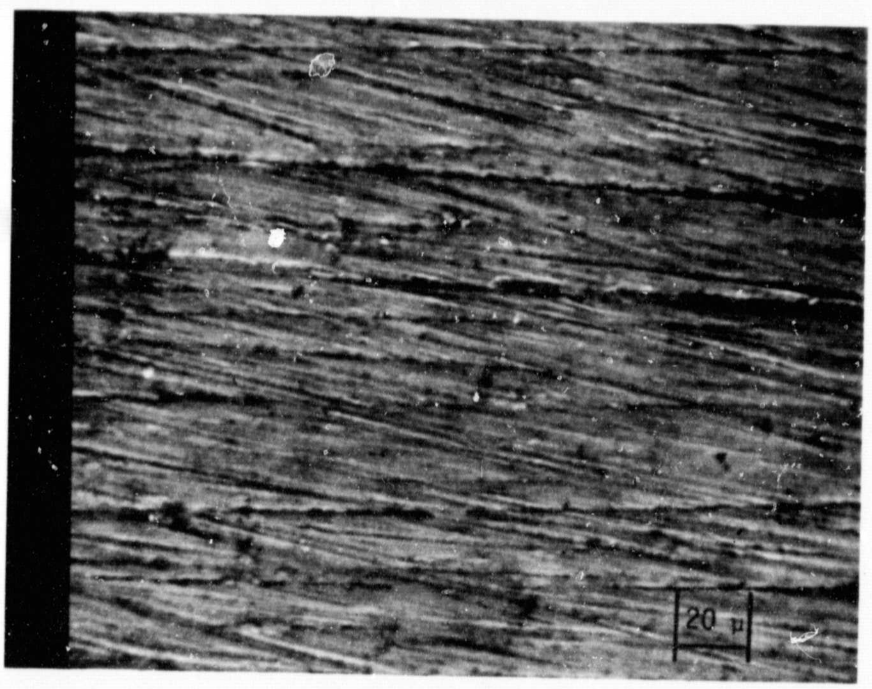


b) Top to bottom: 7552, 7551, 7521, 7502

Figure E-5. Photographs of Passivated Specimens.



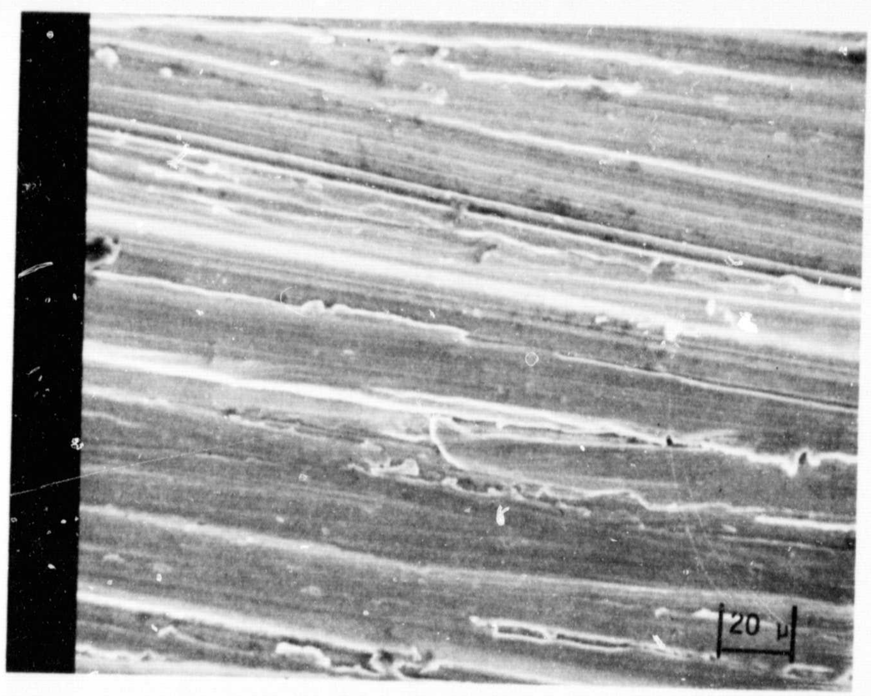
a) 7500, ∿ 250X HT/16



b) 7500, ∿ 500X HT/16

Figure E-6: SEM Photographs of Unpassivated Specimens

c) 7520, ~ 250X A/16



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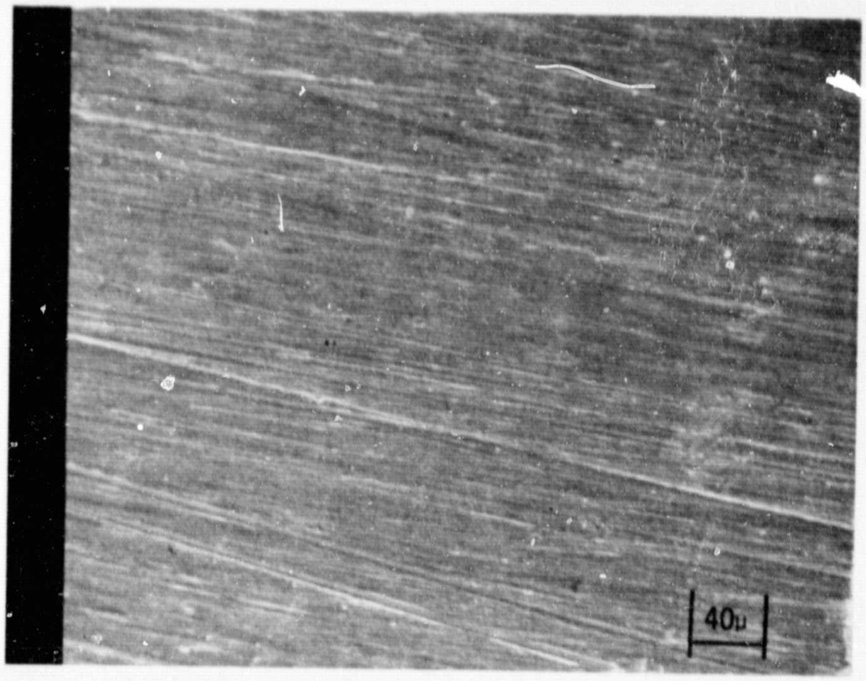

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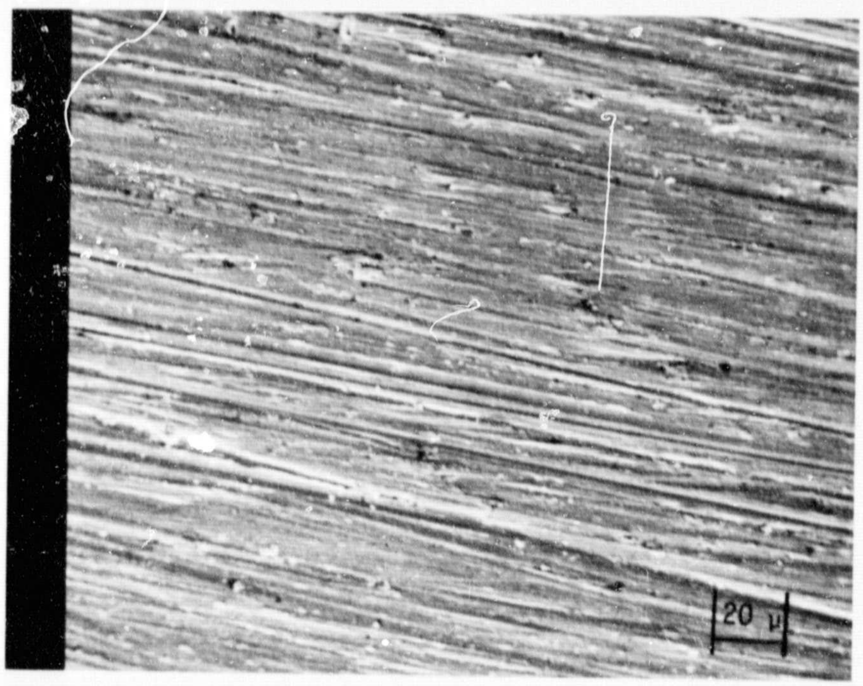

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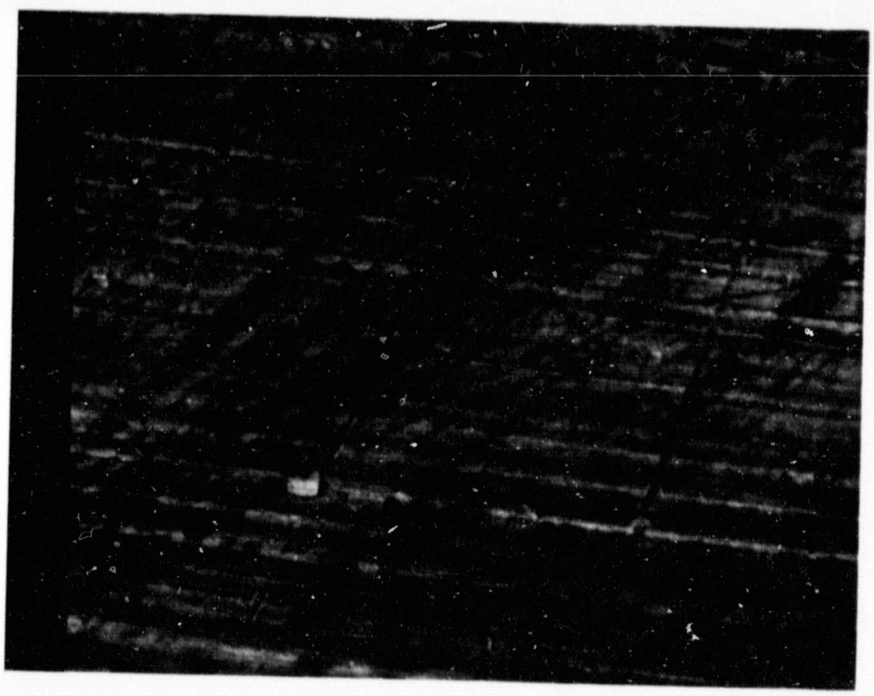

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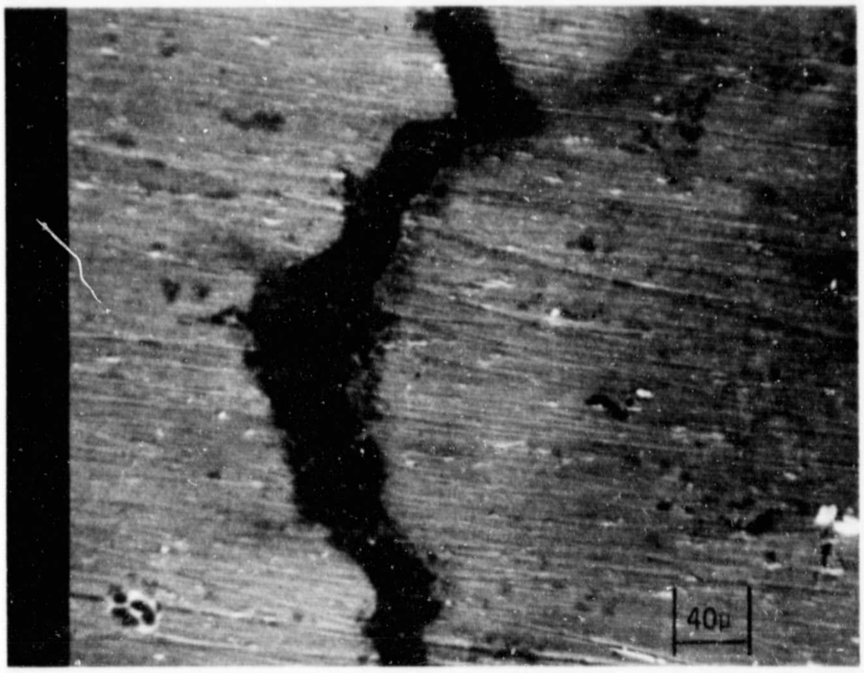
Figure E-6: SEM Photographs of Unpassivated Specimens (Continued)



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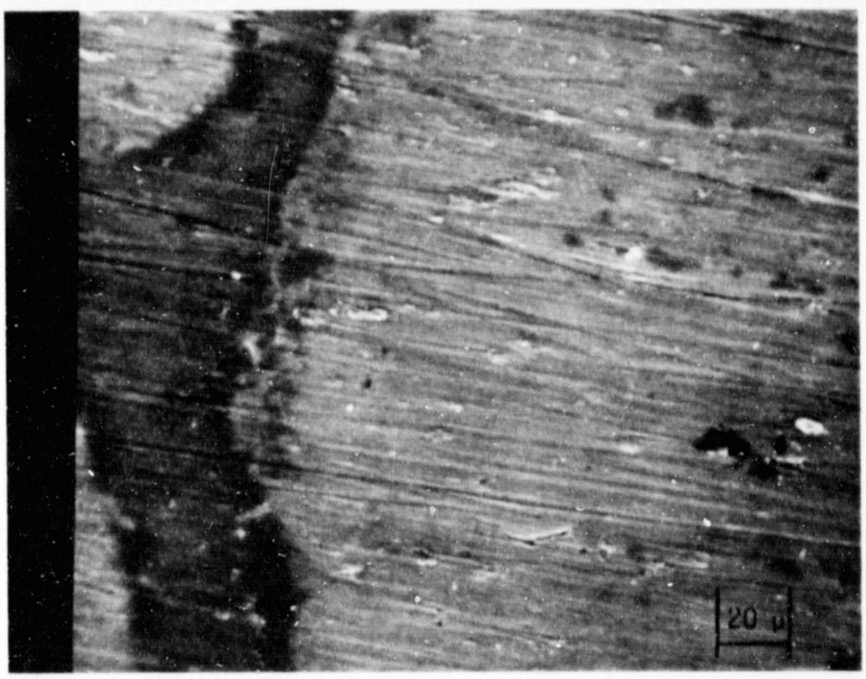

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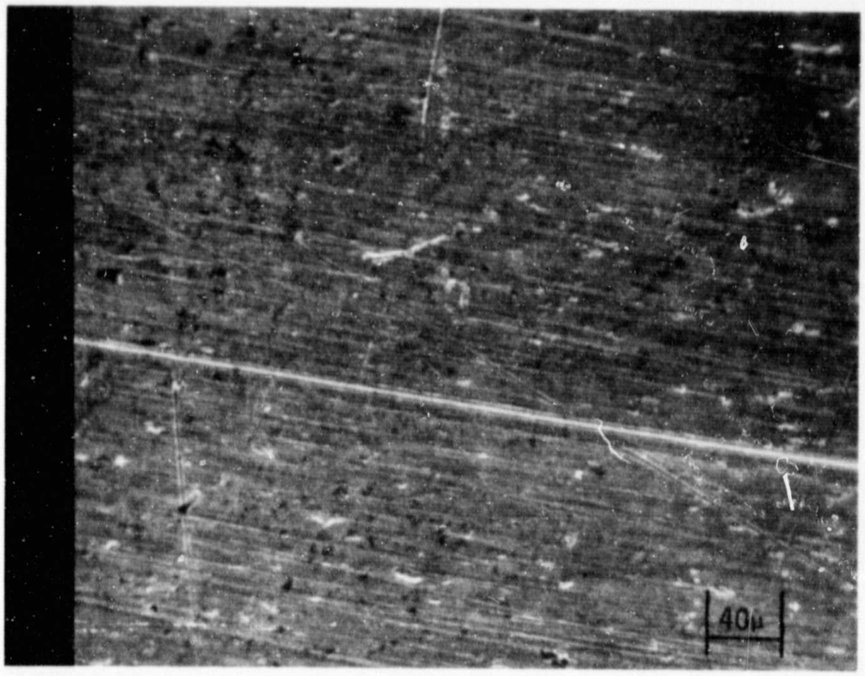
Figure E-7: SEM Photographs of Passivated Specimens



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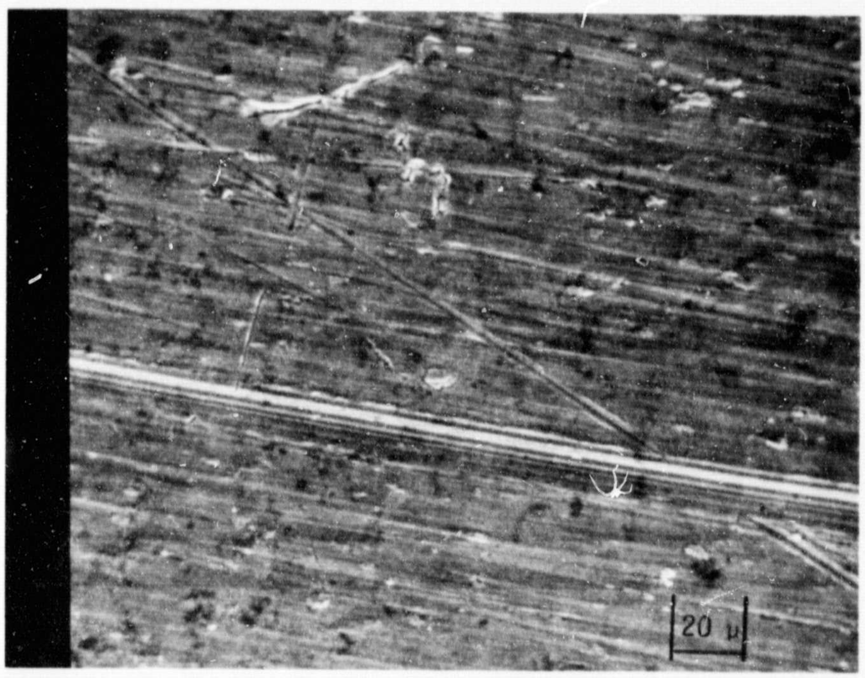

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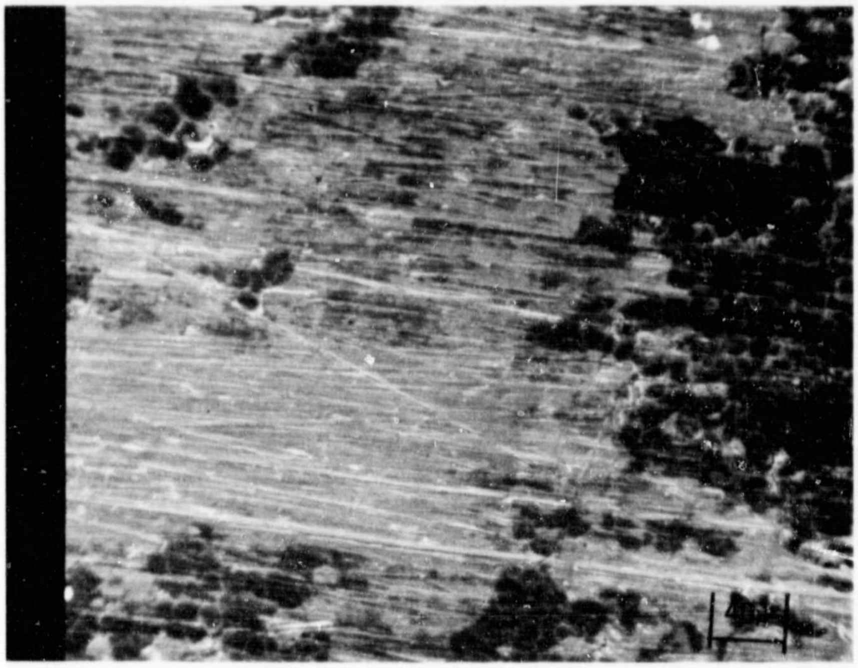

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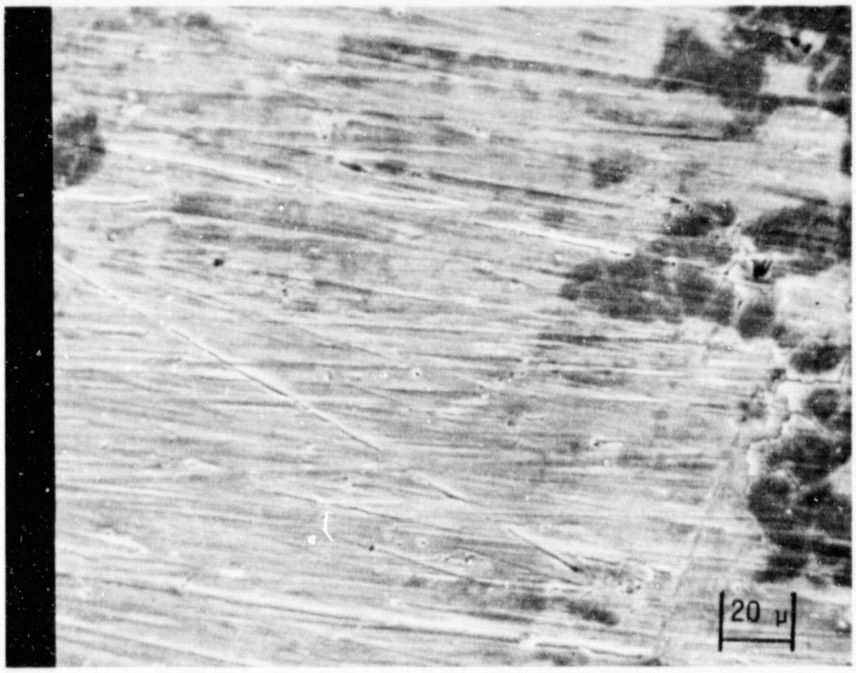

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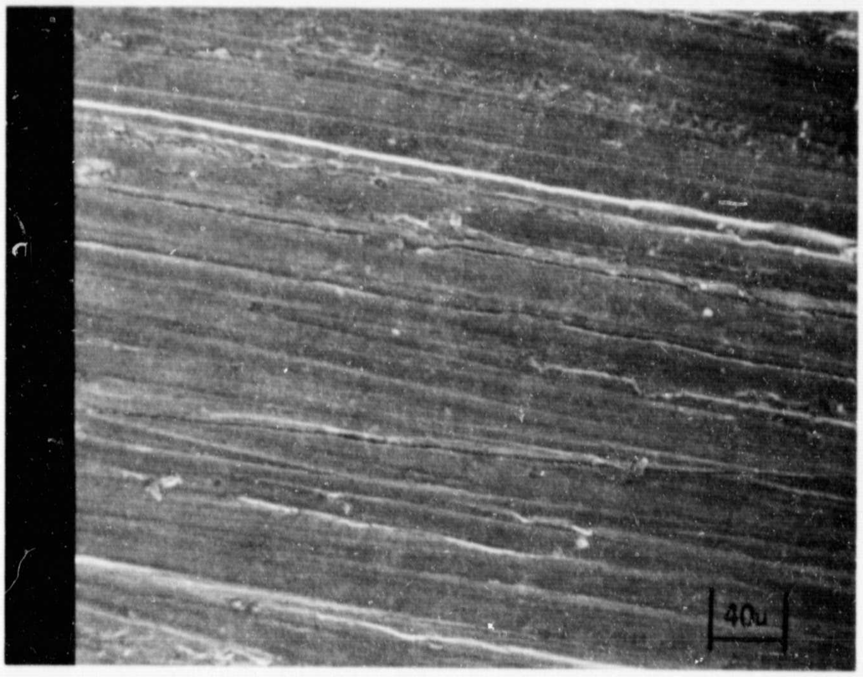
Figure E-7: SEM Photographs of Passivated Specimens (Continued)



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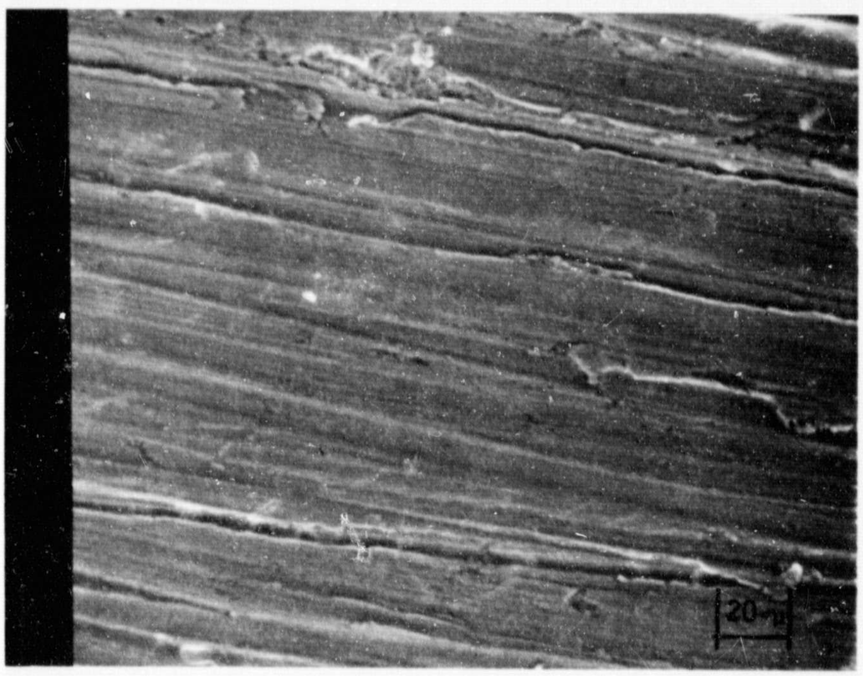

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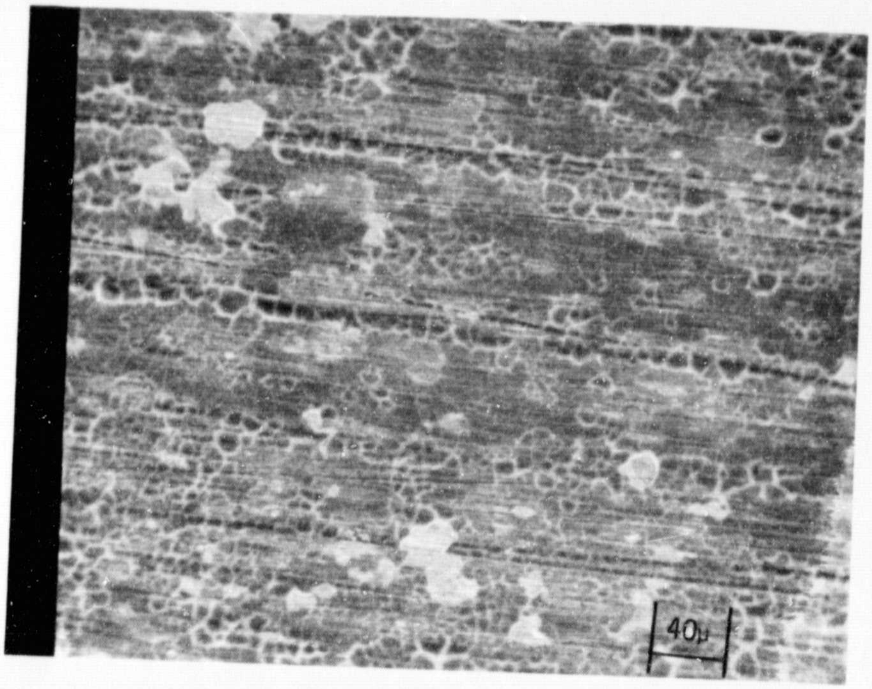
Figure E-7: SEM Photographs of Passivated Specimens (Continued)



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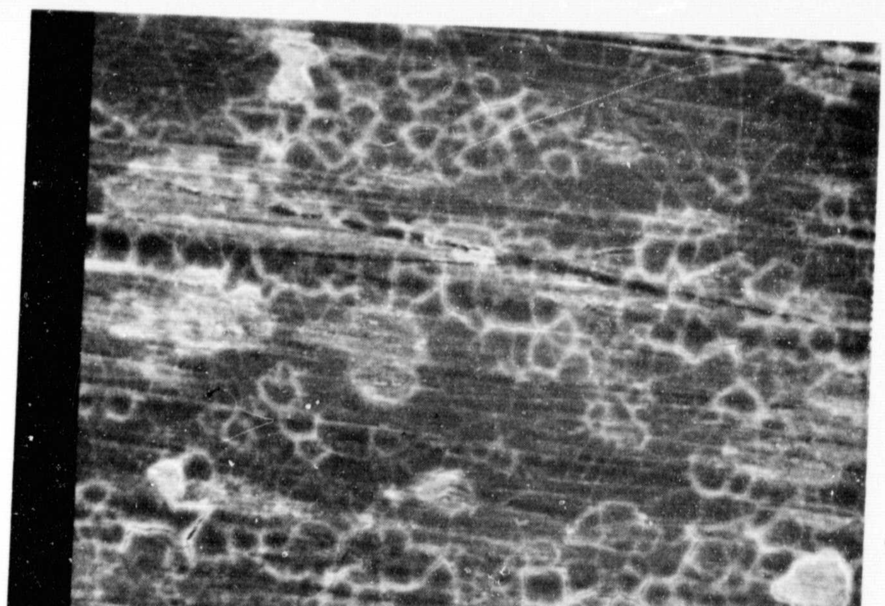

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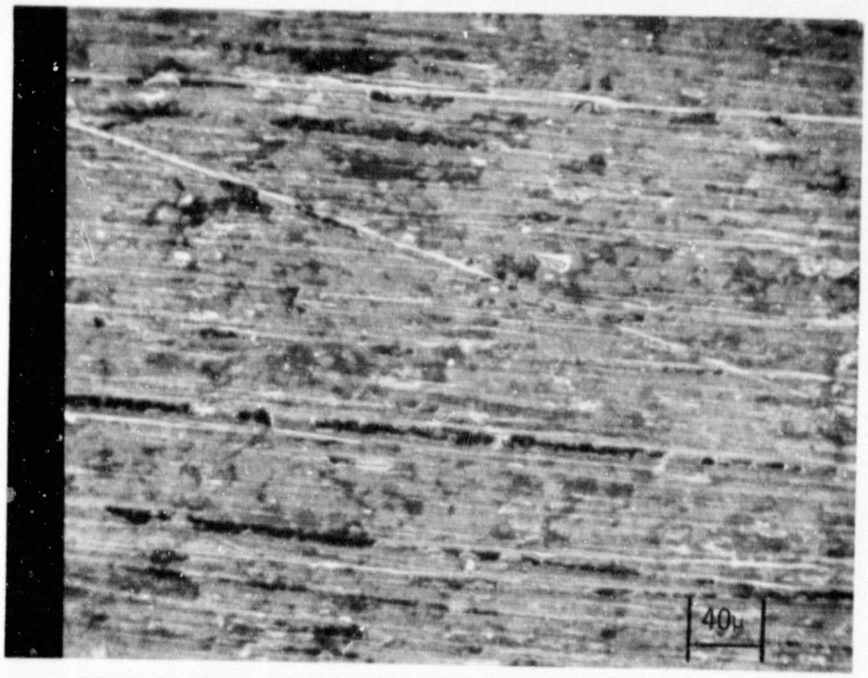
j) 7551, ~ 500X A/16

Figure E-7: (Continued) SEM Photographs of Passivated Specimens



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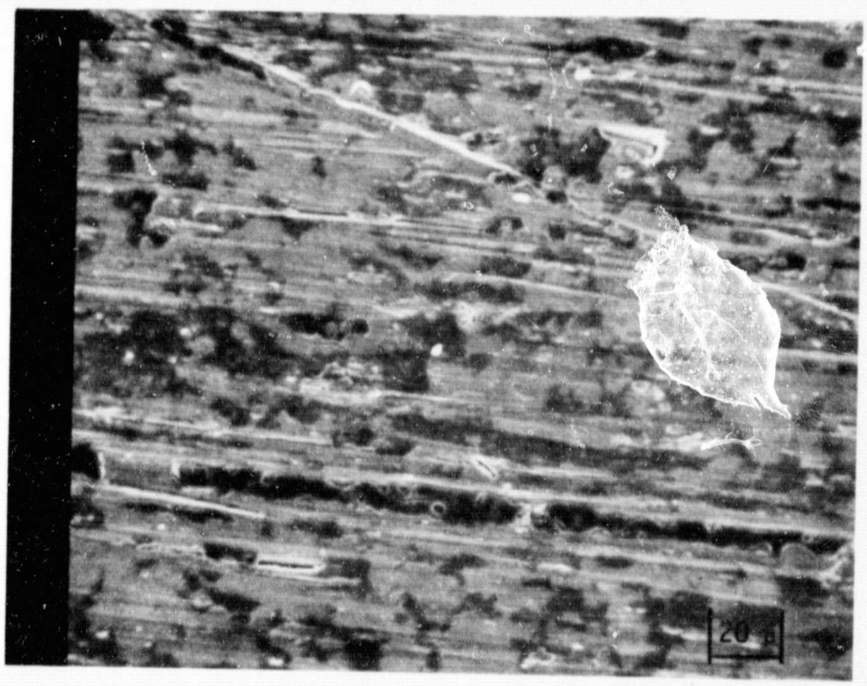

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Figure E-7: SEM Photographs of Passivated Specimens (continued)

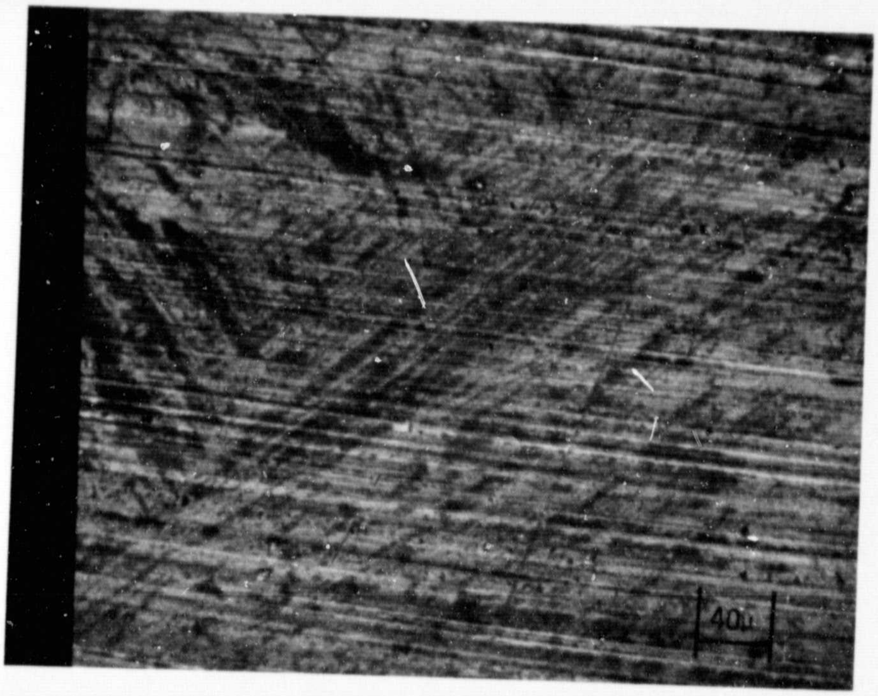

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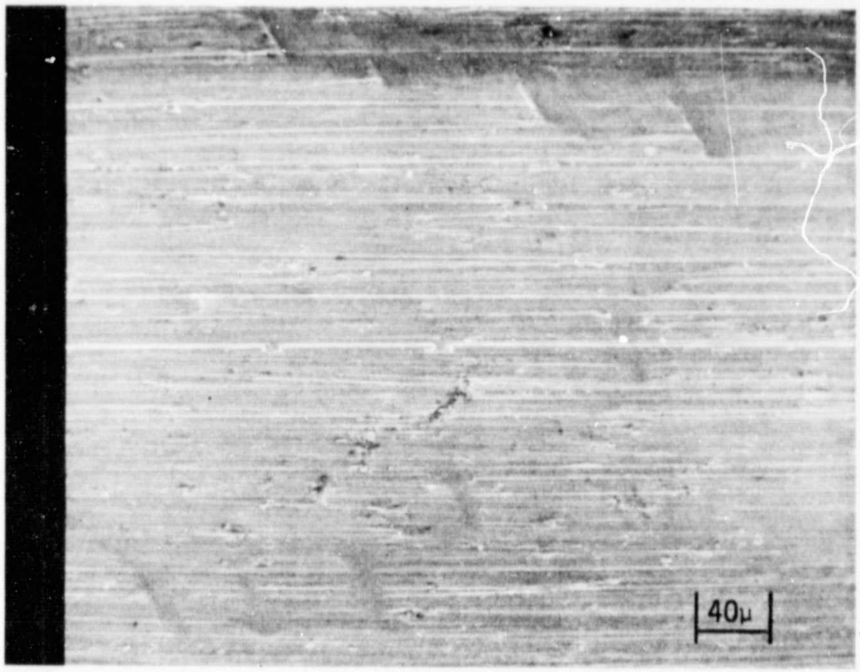
Figure E-7: SEM Photographs of Passivated Specimens (Continued)



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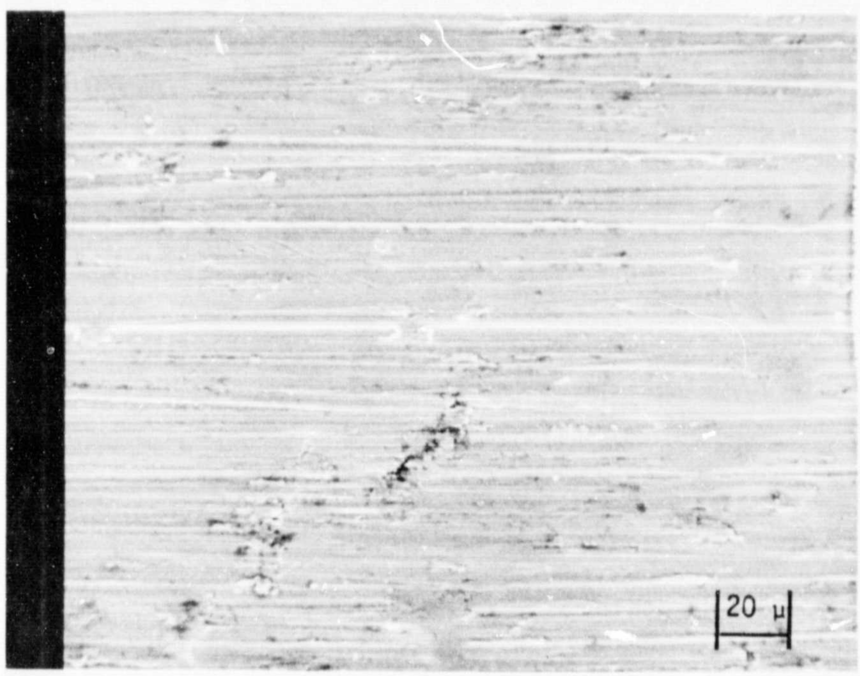

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Figure E-7: SEM Photographs of Passivated Specimens (Continued)

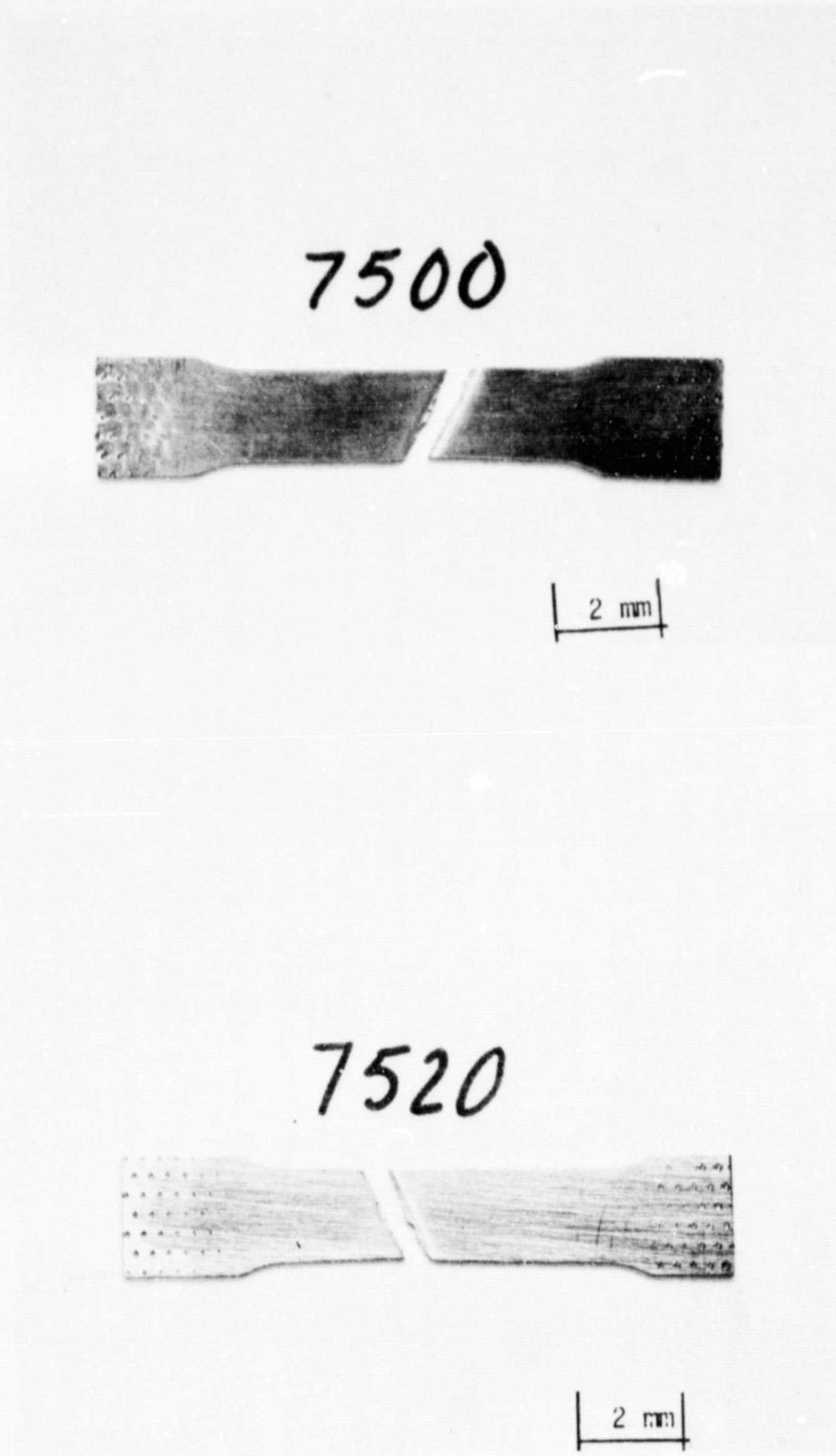
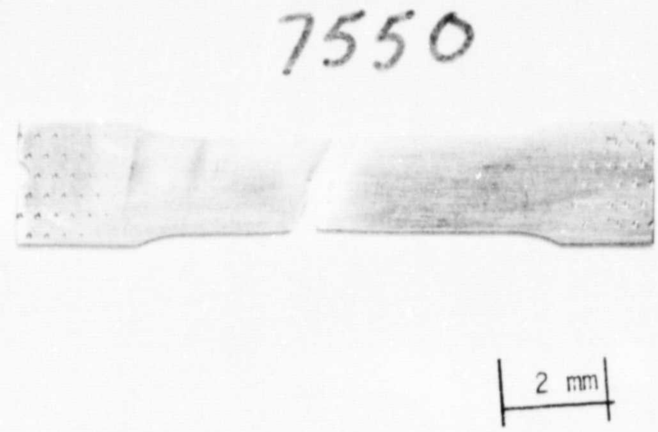


Figure E-8: Photographs of Fractured Tensile Coupons, Unpassivated, 298K



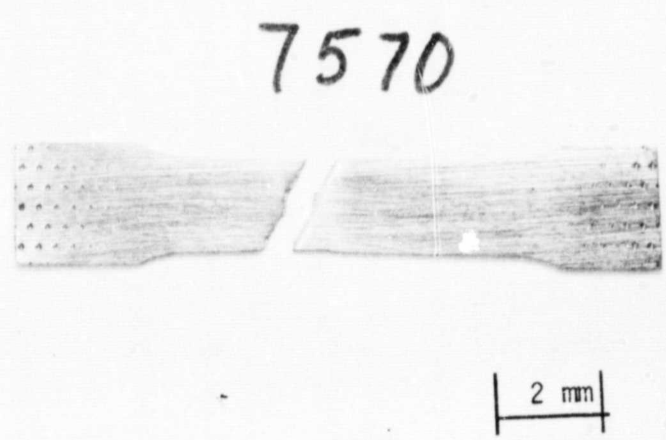


Figure E-8: Photographs of Fractured Tensile Coupons,
(Continued) Unpassivated, 298K

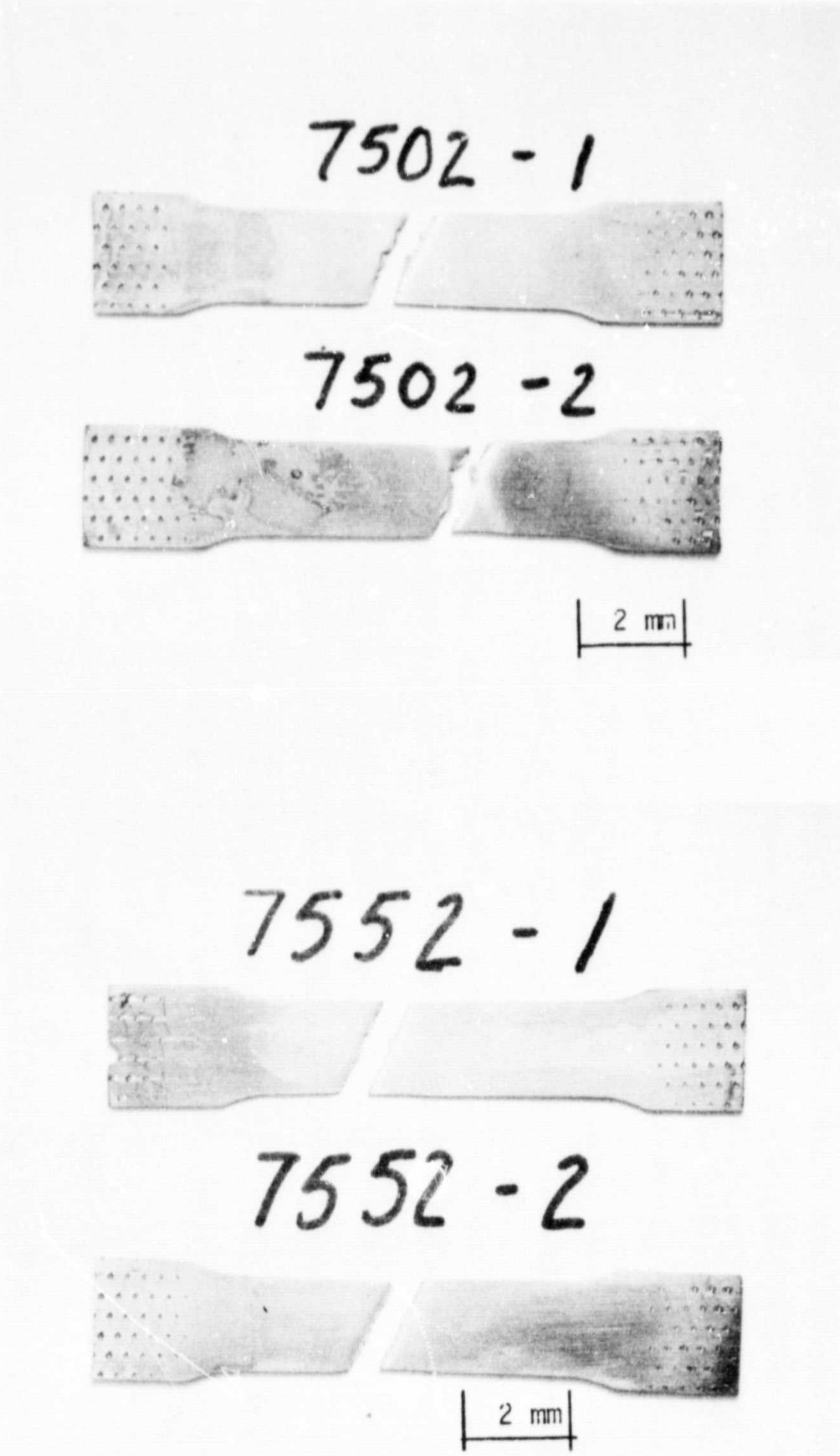


Figure E-9: Photographs of Fractured Tensile Coupons, Passivated, 298K

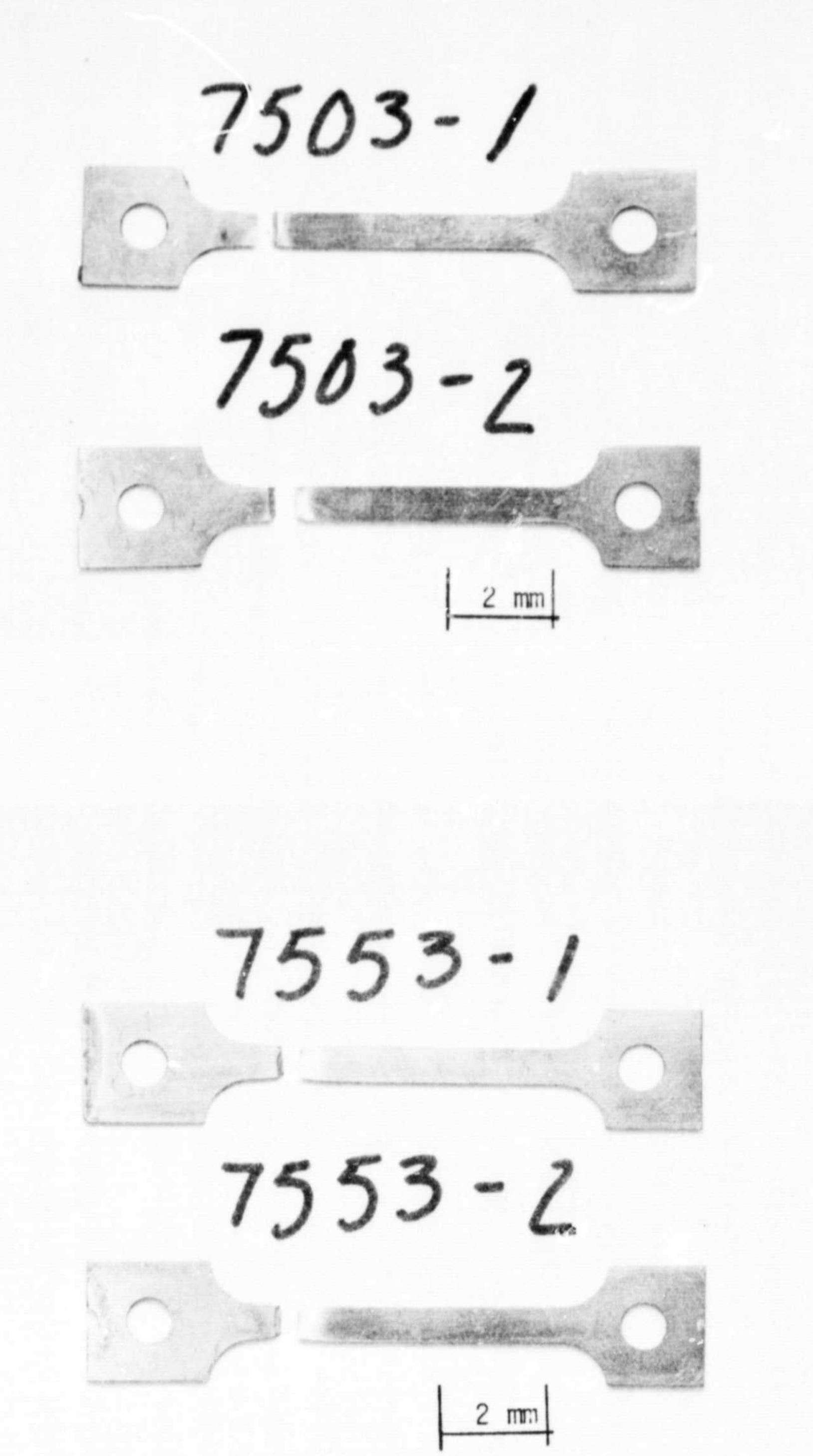


Figure E-10. Photographs of Fractured Tensile Coupons, Passivated, 77K

TABLE E-I: PIT DEPTH FREQUENCY DISTRIBUTION: PRE-IMMERSION COUPONS

	Percent of Pits in Various Average Depth Ranges (10^{-4}cm)						
Specimen No.	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7
Range Mean	0.01-0.64 (0.325)	0.65-1.28 (0.965)	1.29-1.92 (1.61)	1.93-2.56 (2.24)	2.57-3.20 (2.89)	3.21-3.84 (3.42)	3.85-4.48 (4.16)
7500	11.1	88.9	0	0	0	0	0
7520	0	38.9	11.1	27.8	16.7	0	5.6
7550	0	40.0	0	30.0	30.0	0	0
7570	0	33.3	11.1	33.3	11.1	0	11.1
7501	15.4	61.5	0	15.4	7.7	0	0
7502	35.7	57.1	7.1	0	0	0	0
7503	12.5	50.0	6.25	31.25	0	0	0
7521	0	81.8	0	9.1	9.1	0	0
7551	0	60.0	0	40.0	0	0	0
7552	15.4	69.2	0	15.4	0	0	0
7553	0	90.0	0	10.0	0	0	0
7571	0	66.7	0	22.2	11.1	0	0

TABLE E-II: PIT AREA FREQUENCY DISTRIBUTION: PRE-IMMERSION COUPONS

	Percent of Pits in Various Average Area Ranges ($10^{-7} cm^2$)						
Specimen No.	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7
Range Mean	0.001-0.049 (0.025)	0.050-0.099 (0.0745)	0.100-0.490 (0.295)	0.500-0.990 (0.745)	1.000-4.999 (3.000)	5.000-9.999 (7.500)	10.000-14.999 (12.500)
7500	22.2	22.2	11.1	33.3	11.1	0	0
7520	5.6	11.1	11.1	27.8	27.8	5.6	11.1
7550	0	0	10.0	30.0	50.0	10.0	0
7570	11.1	0	22.2	22.2	33.3	11.1	0
7501	15.4	23.1	15.4	15.4	30.8	0	0
7502	0	0	28.6	42.9	28.6	0	0
7503	0	0	12.5	25.0	50.0	6.2	6.2
7521	18.2	9.1	54.5	18.2	0	0	0
7551	0	0	40.0	40.0	20.0	0	0
7552	15.4	7.7	38.5	15.4	23.1	0	0
7553	0	10.0	60.0	30.0	0	0	0
7571	0	0	55.6	33.3	11.1	0	0

TABLE E-III: PIT DEPTH/DIAMETER RATIO FREQUENCY DISTRIBUTION: PRE-IMMERSION COUPONS

	Percent of Pits with Various Average Depth/Diameter ($\frac{1}{d}$) Ratios						
Specimen No.	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7
Range Mean	0.01-0.20 (0.105)	0.21-0.41 (0.31)	0.42-0.52 (0.52)	0.63-0.83 (0.73)	0.84-1.04 (0.94)	1.05-1.25 (1.15)	1.26-2.00 (1.63)
7500	0	11	44	11	33	0	0
7520	0	11	44	11	22	11	0
7550	0	0	50	40	10	0	0
7570	0	0	22	22	56	0	0
7501	0	8	31	23	31	7	0
7502	21	57	22	0	0	0	0
7503	25	19	44	12	0	0	0
7521	0	0	18	18	36	0	27
7551	0	0	20	20	60	0	0
7552	0	15	38	23	23	0	0
7553	0	0	30	50	20	0	0
7571	0	0	22	33	45	0	0

APPENDIX F

APPEARANCE OF 29-MONTH IMMERSION COUPONS

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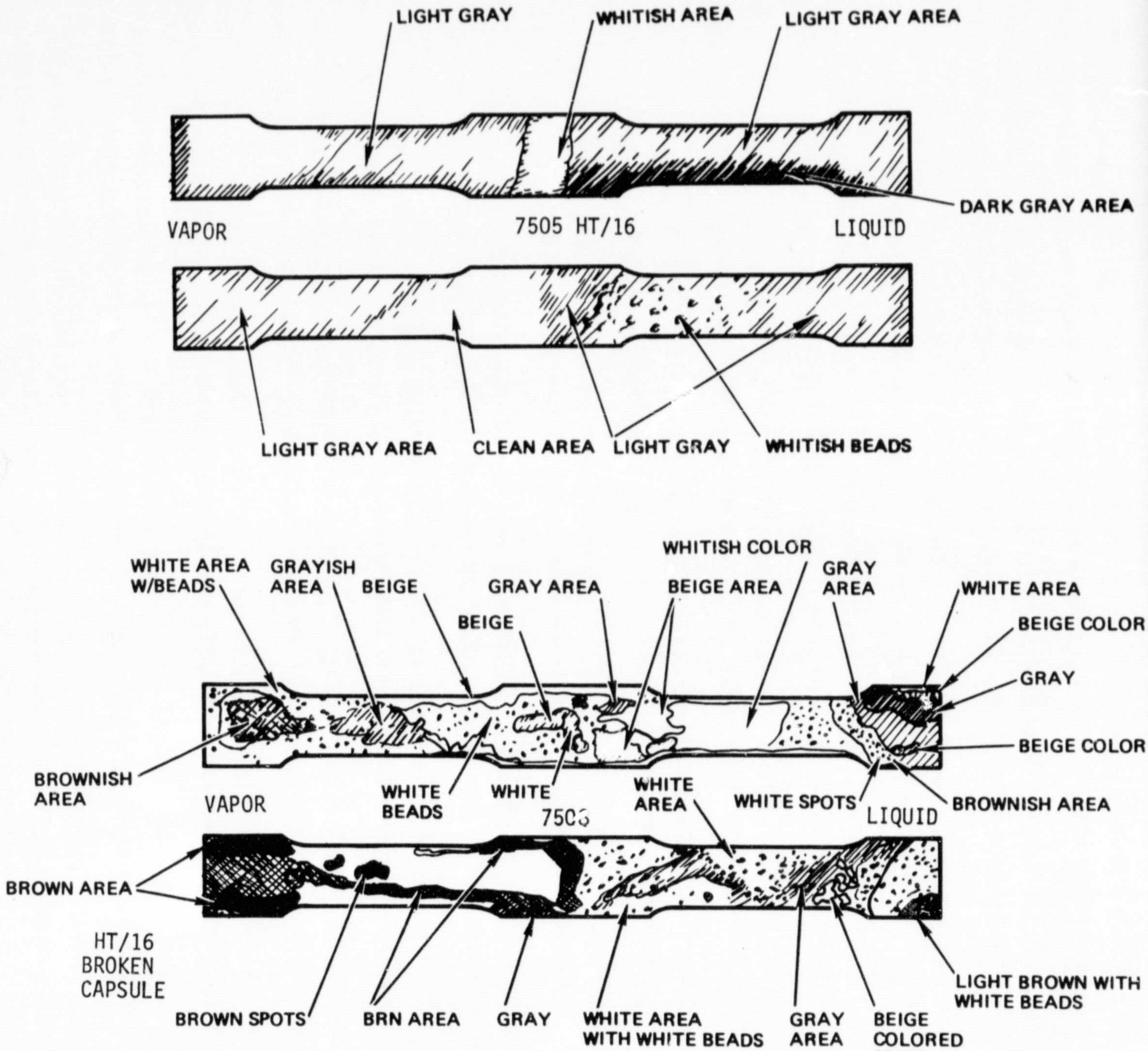


Figure F-1: Maps of the Surfaces of 29-Month Immersion Specimens

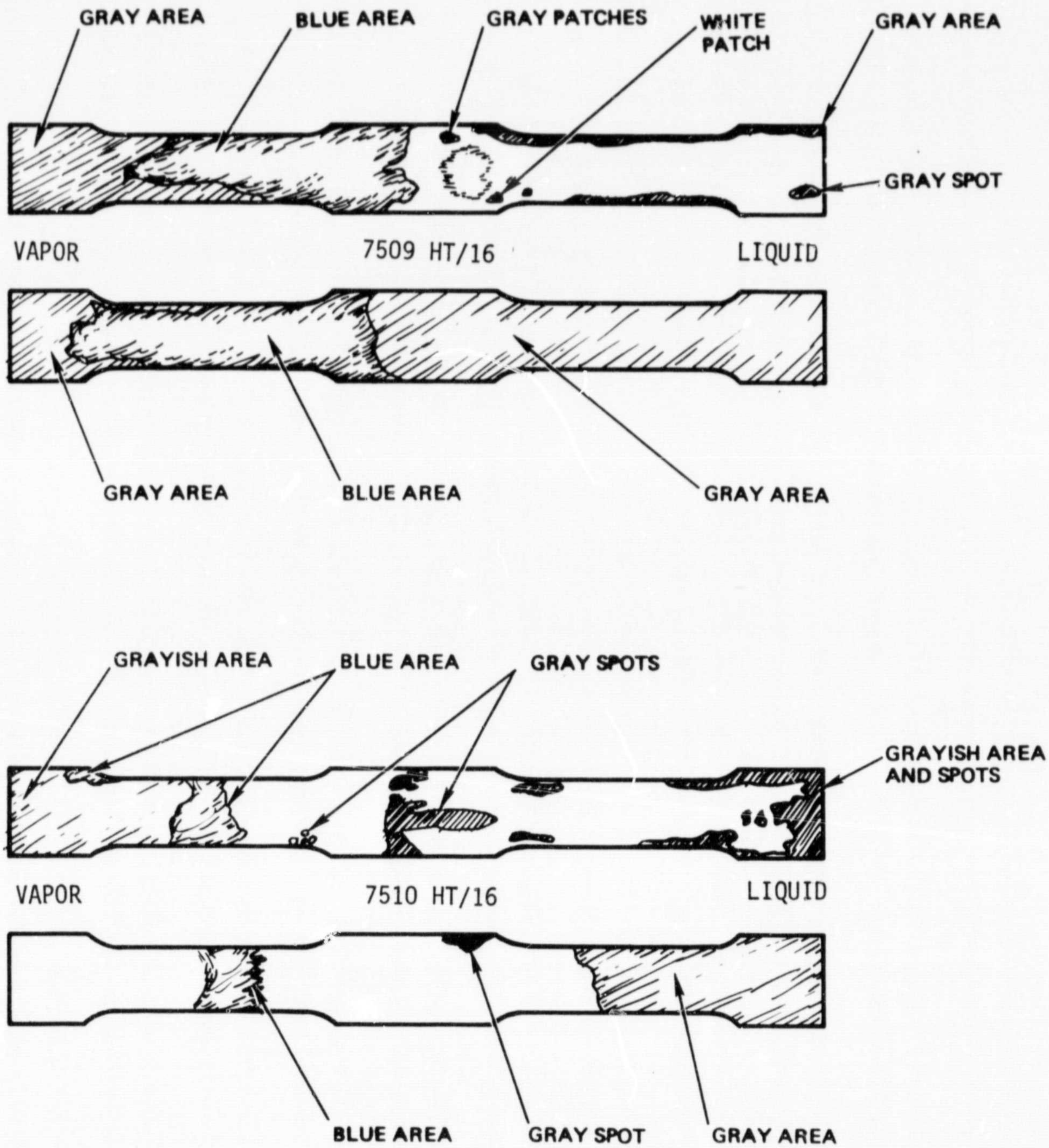


Figure F-1: Maps of the Surfaces of 29-Month
(continued) Immersion Specimens

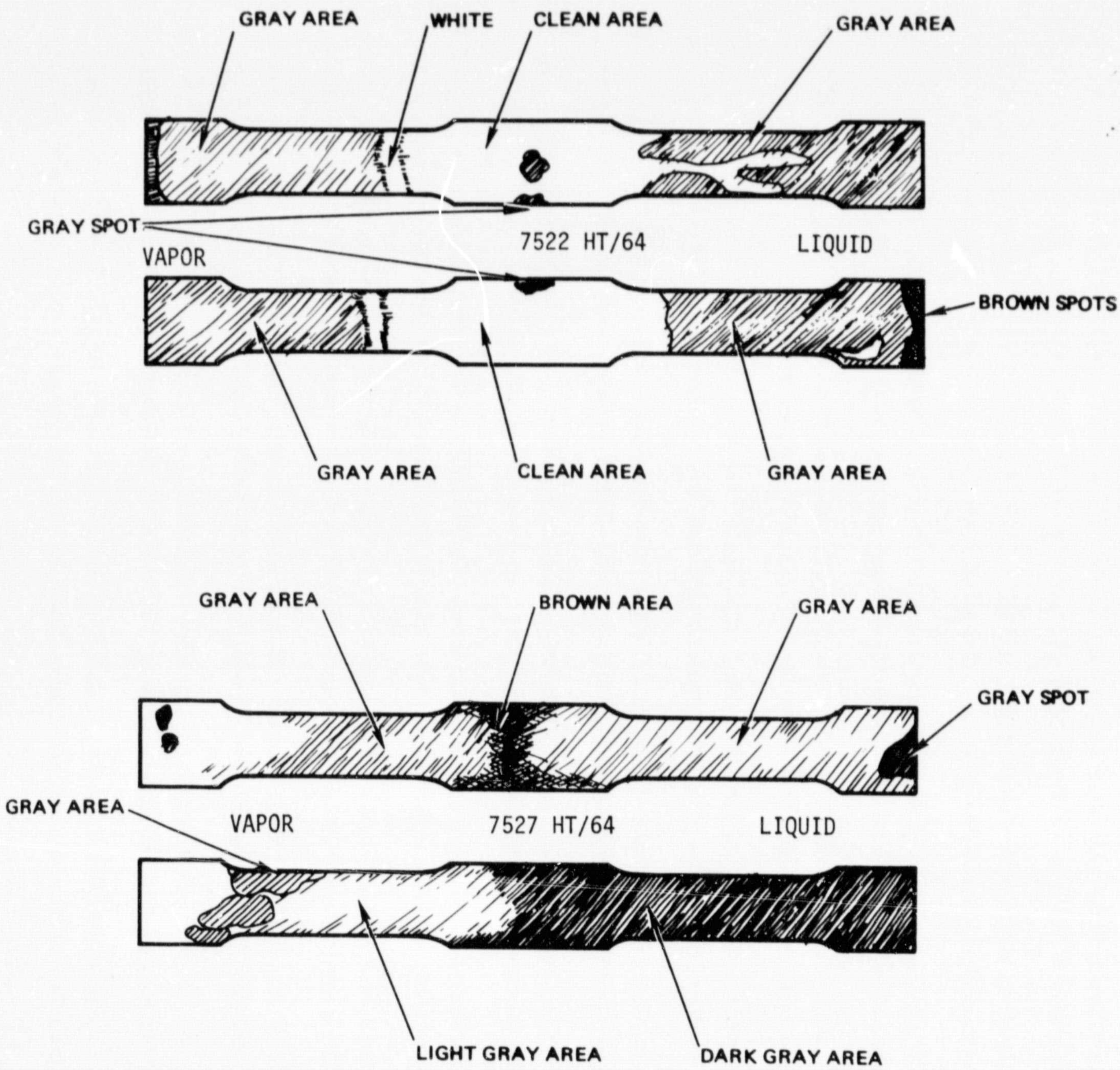


Figure F-1: Maps of the Surfaces of 29-Month
(continued) Immersion Specimens

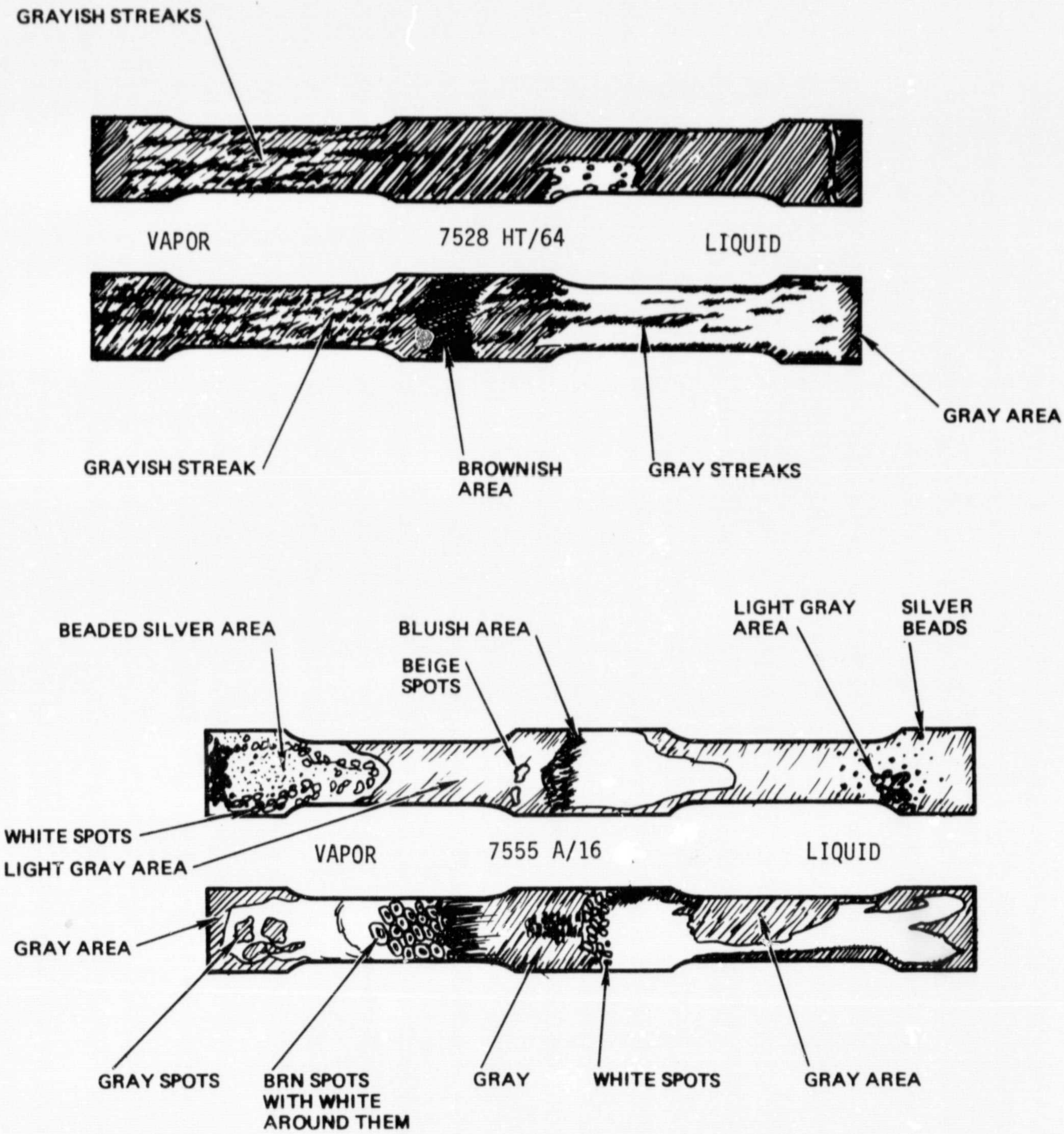


Figure F-1: Maps of the Surfaces of 29-Month
(continued) Immersion Specimens

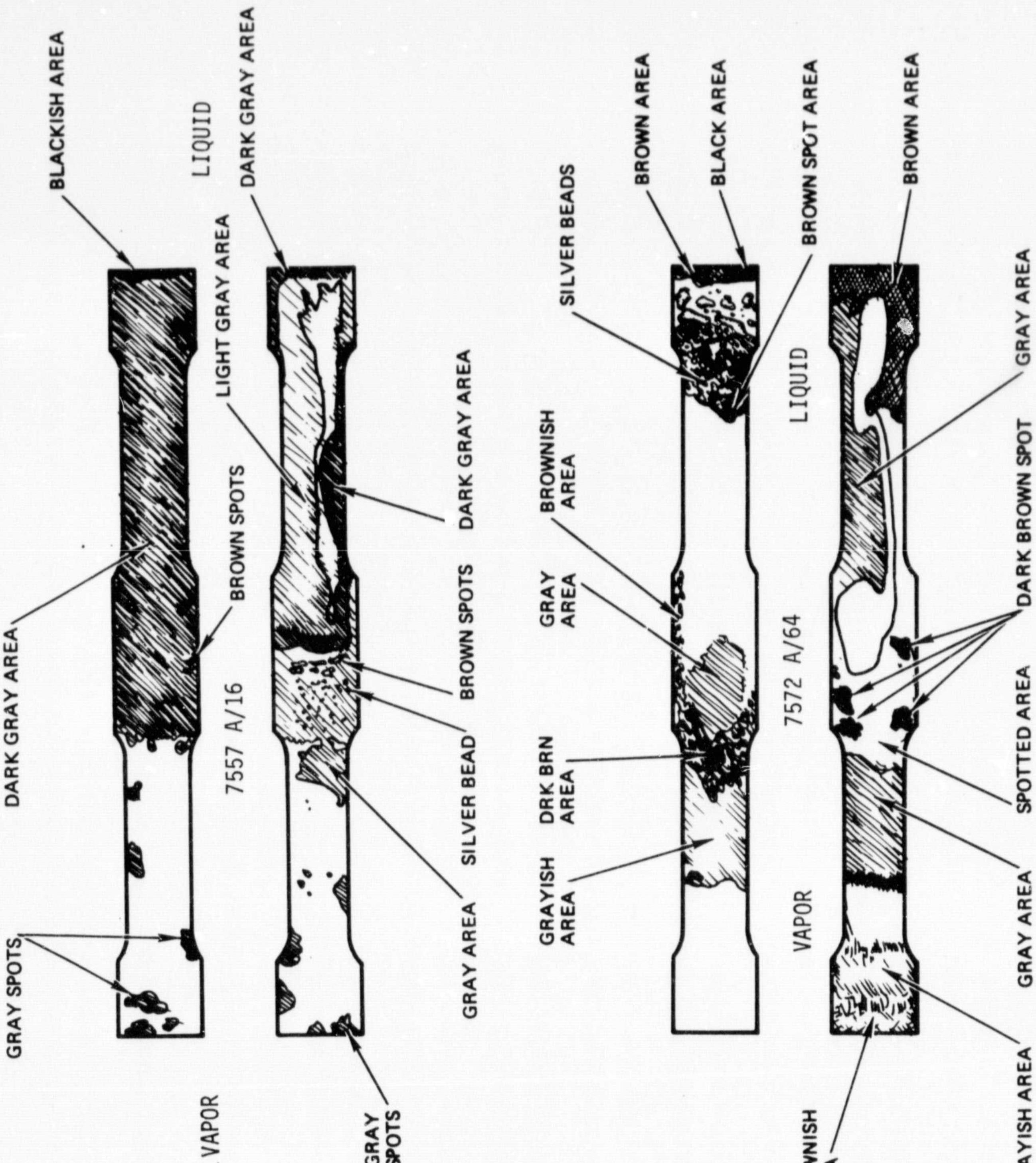


Figure F-1: Maps of the Surfaces of 29-Month
(continued) Immersion Specimens

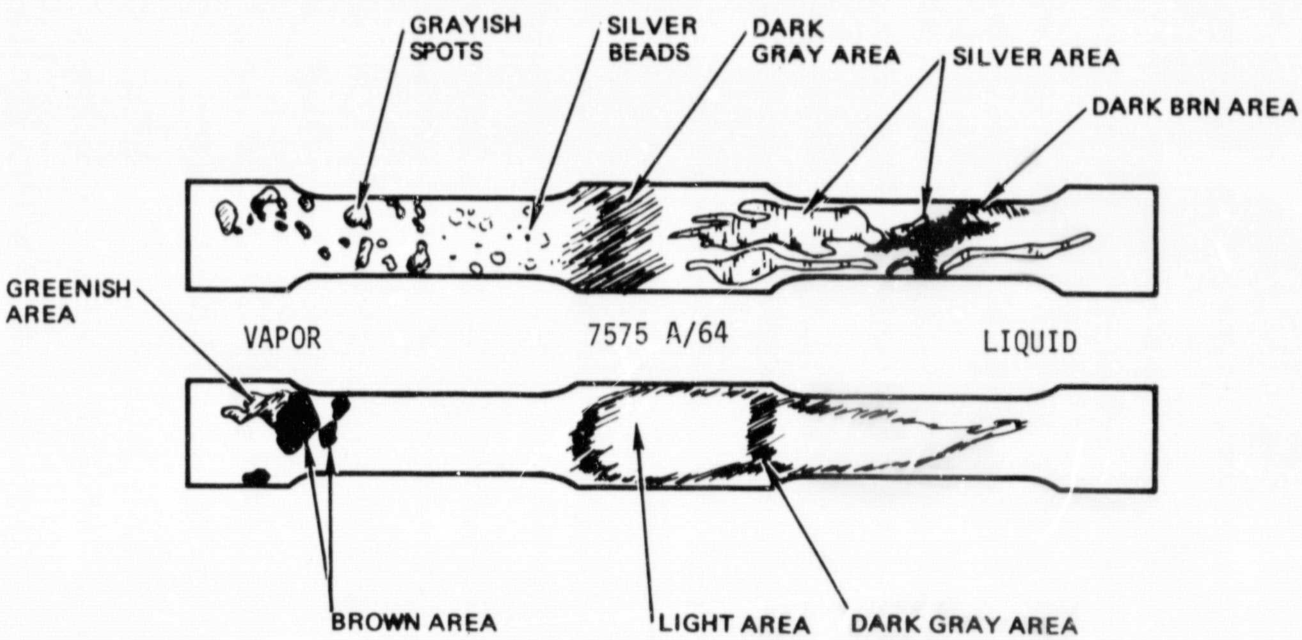


Figure F-1: Maps of the Surfaces of 29-Month
(continued) Immersion Specimens

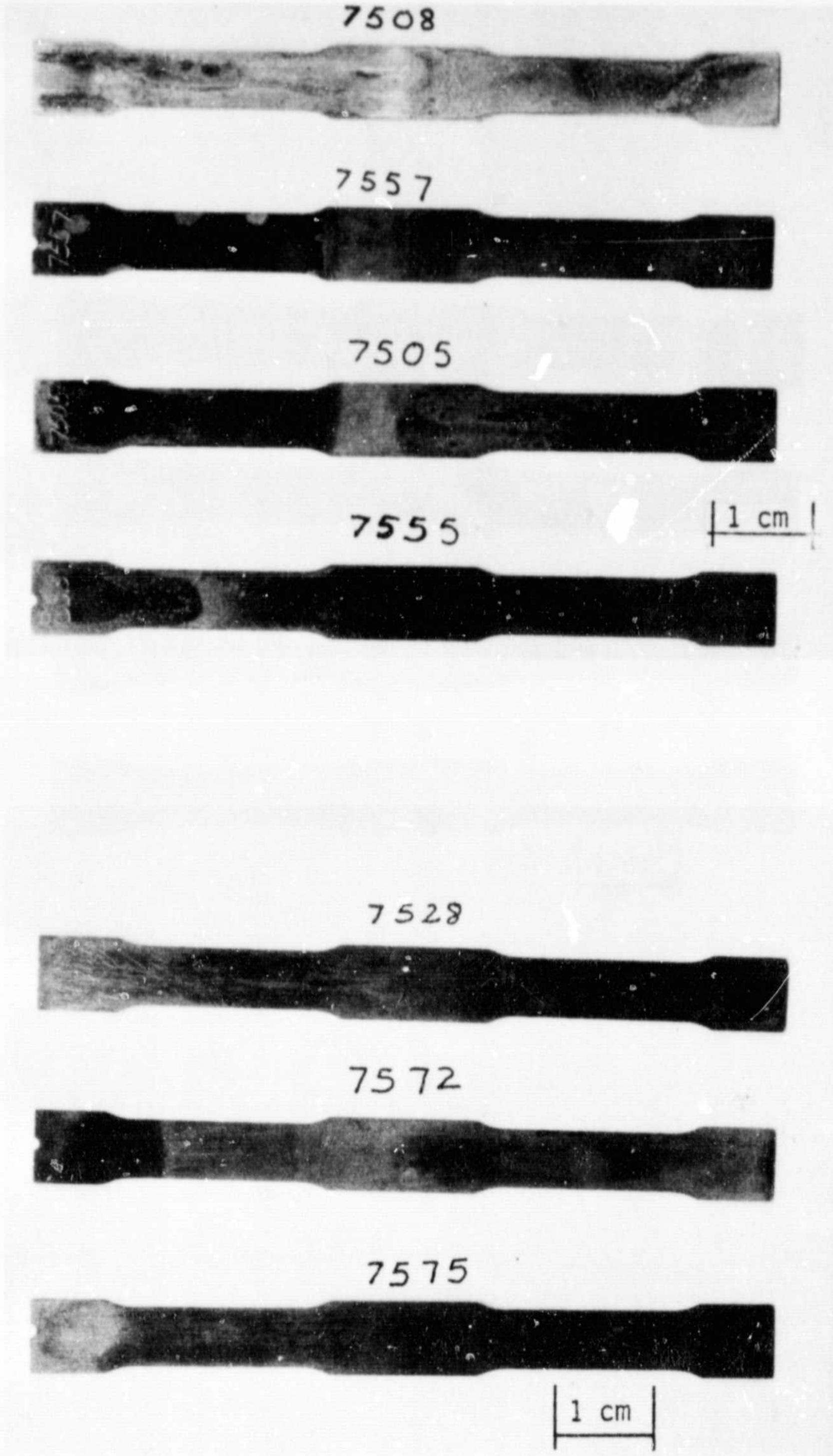


Figure F-2: Photographs of Post-Immersion 29-Month Specimens

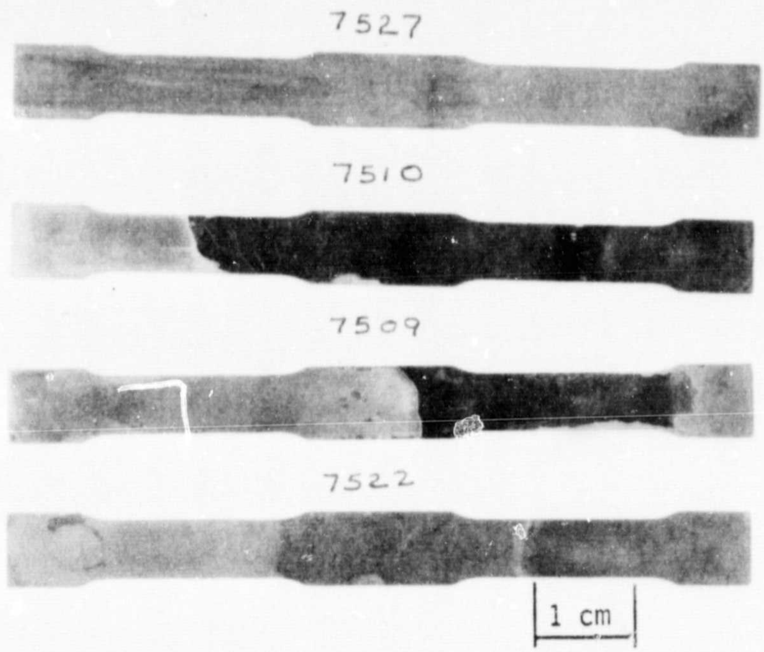
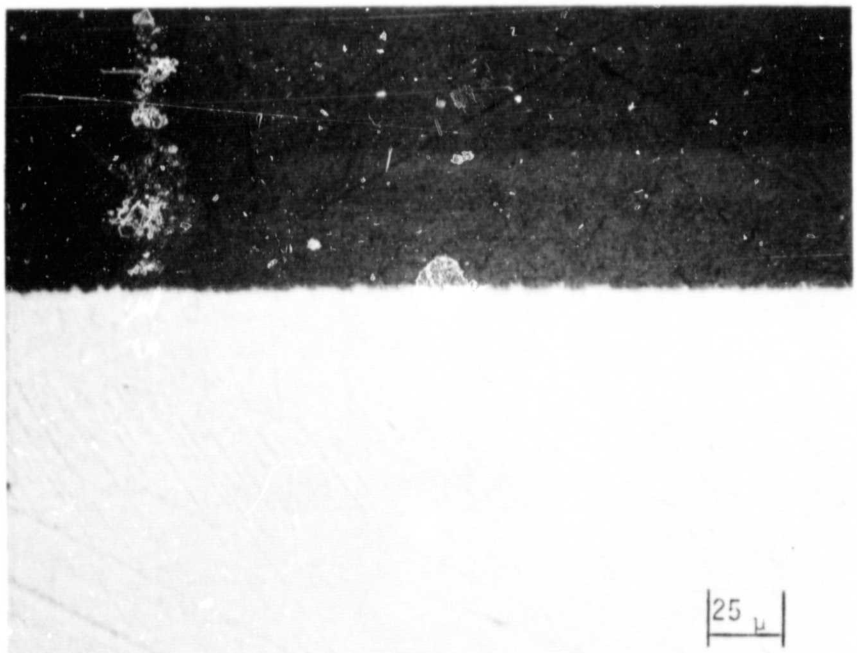
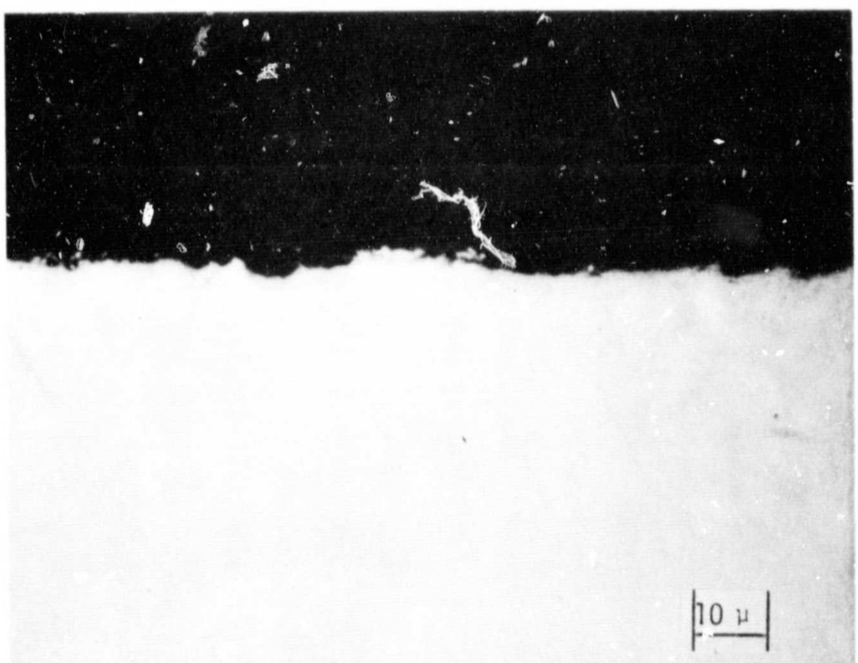


Figure F-2: Photographs of Post-Immersion
(continued) 29-Month Specimens

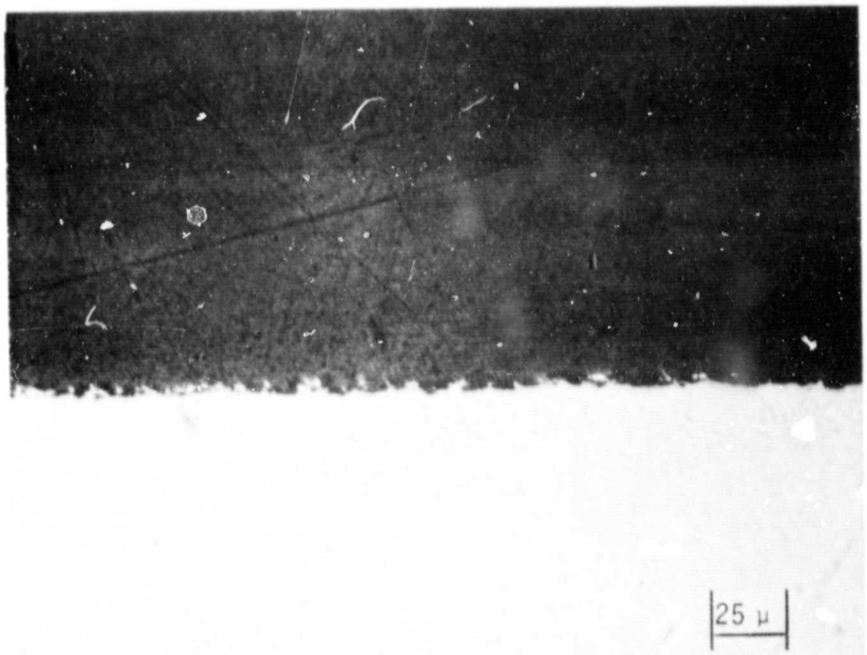


a) 7505, Vapor, ∿ 400X
(Non-typical area)



b) 7505, Vapor, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion 29-Month Specimens



c) 7505, Liquid, ∿ 400X
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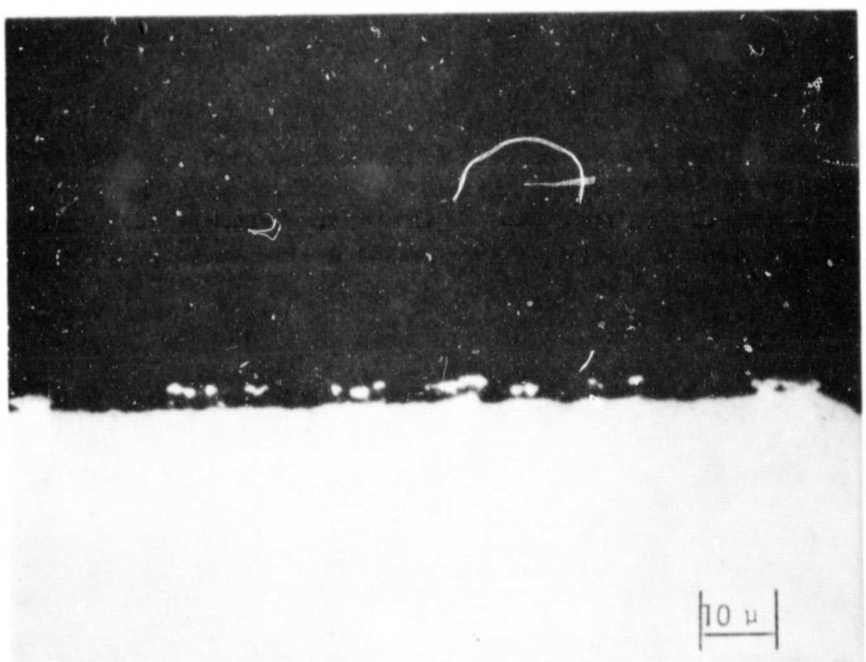
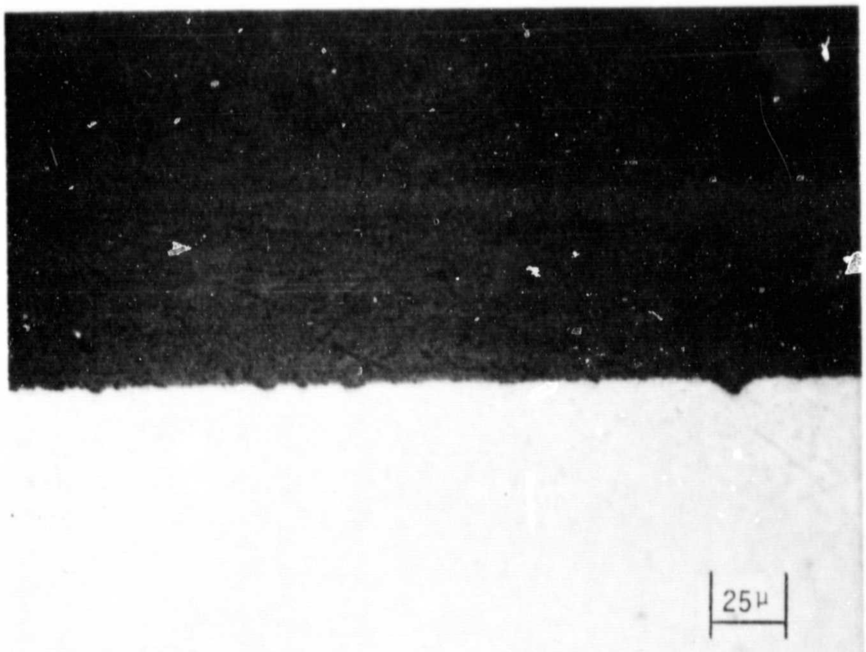
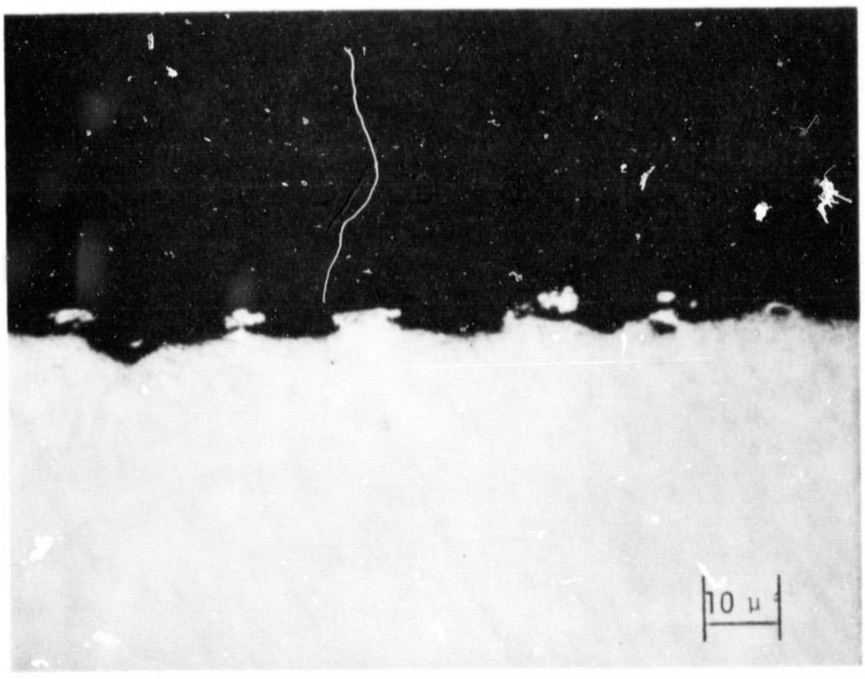


d) 7505, Liquid, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

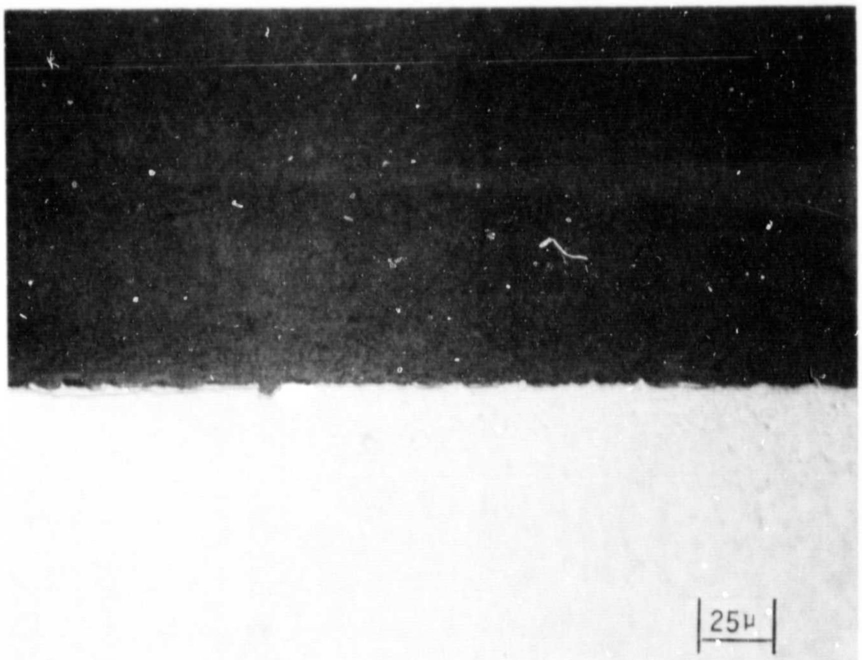


e) 7510, Vapor, ∿ 400X

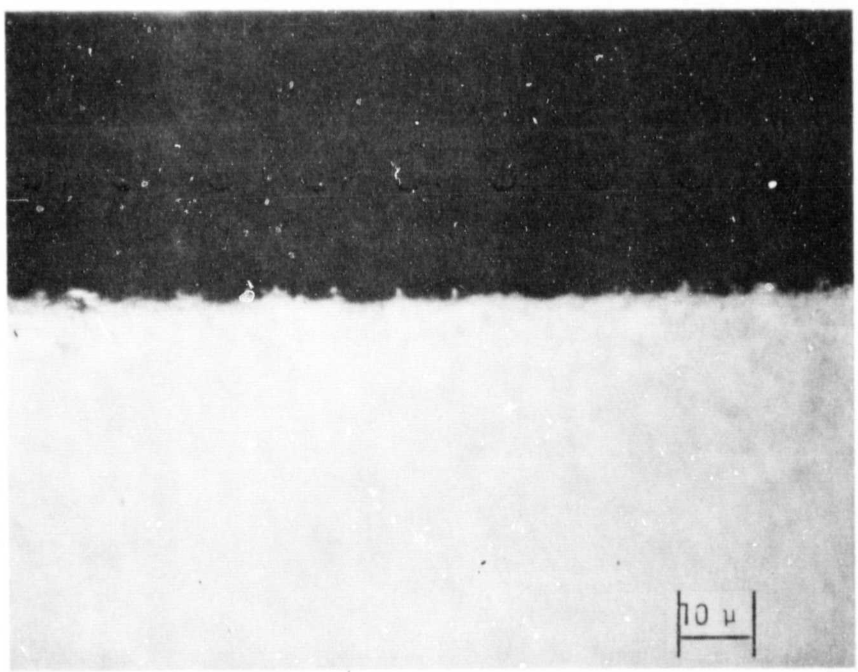


f) 7510, Vapor, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

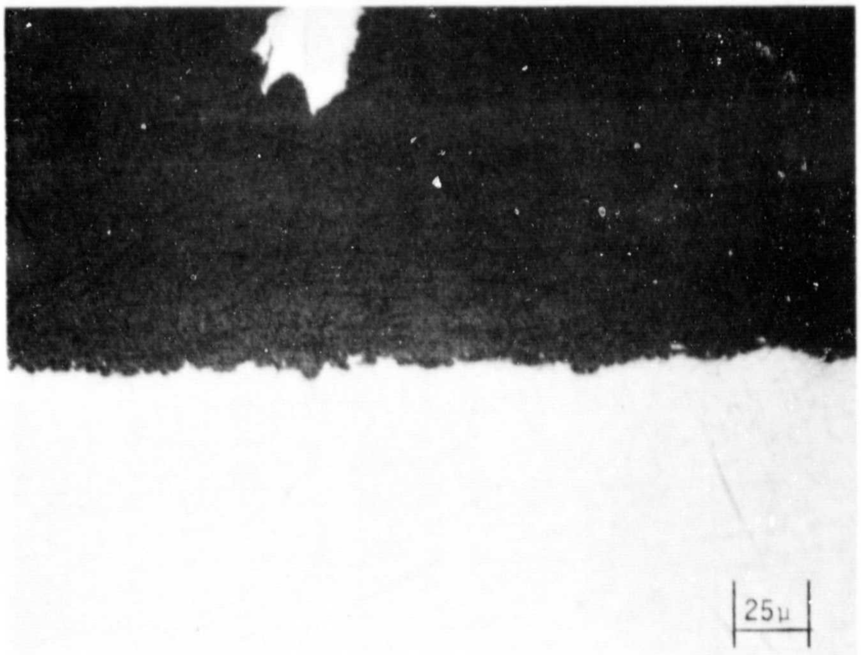


g) 7510, Liquid, ~ 400X

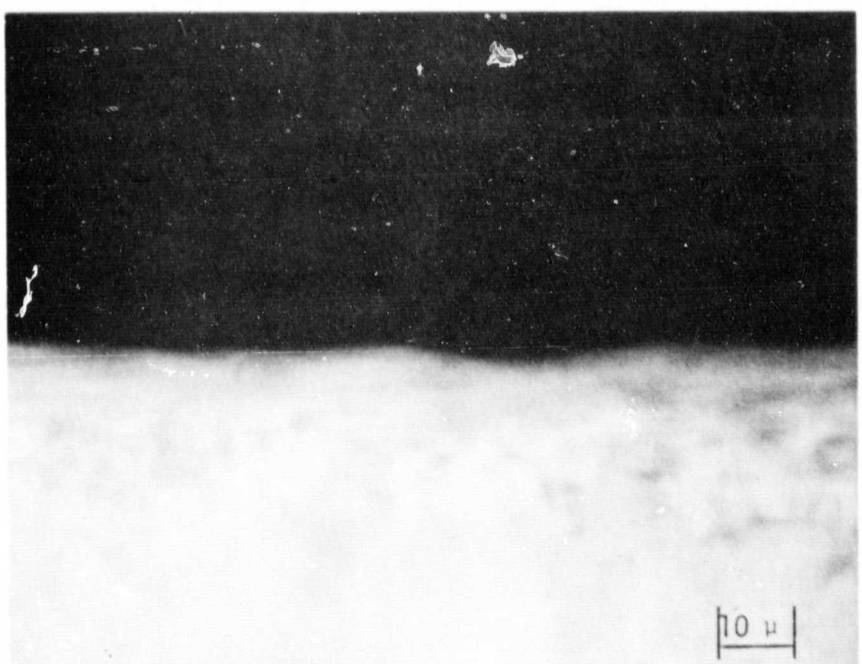


h) 7510, Liquid, ~ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

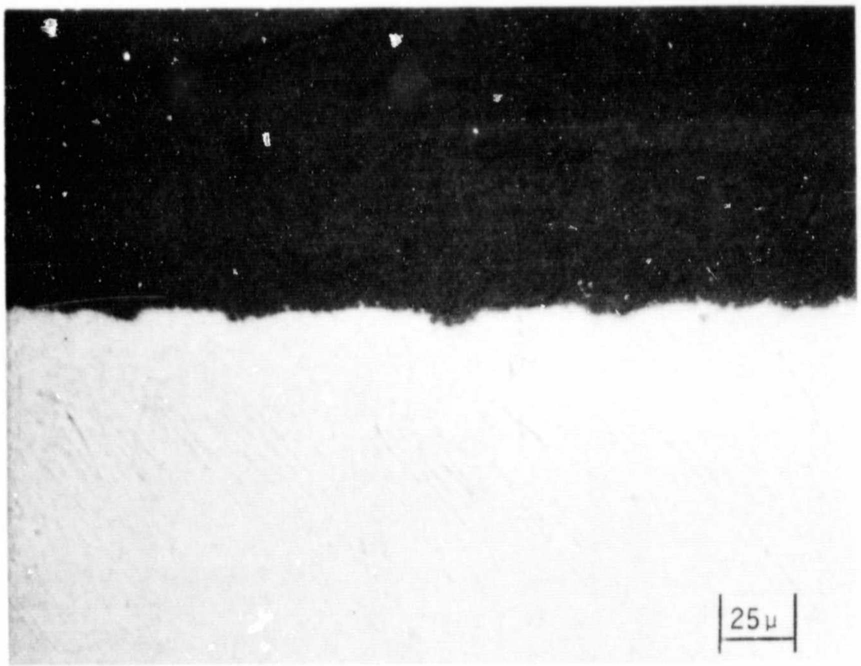


i) 7527, Vapor, ∿ 400X

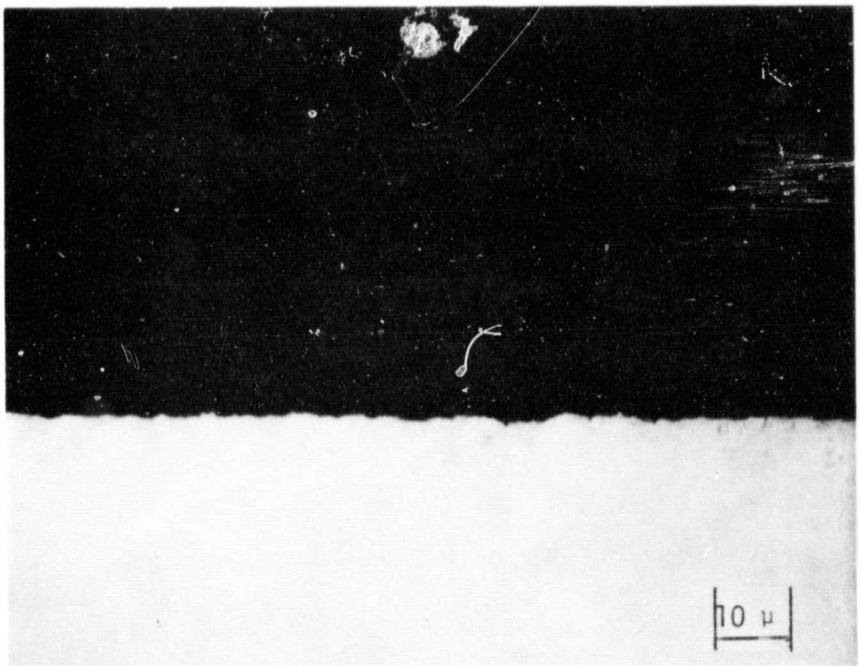


j) 7527, Vapor, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

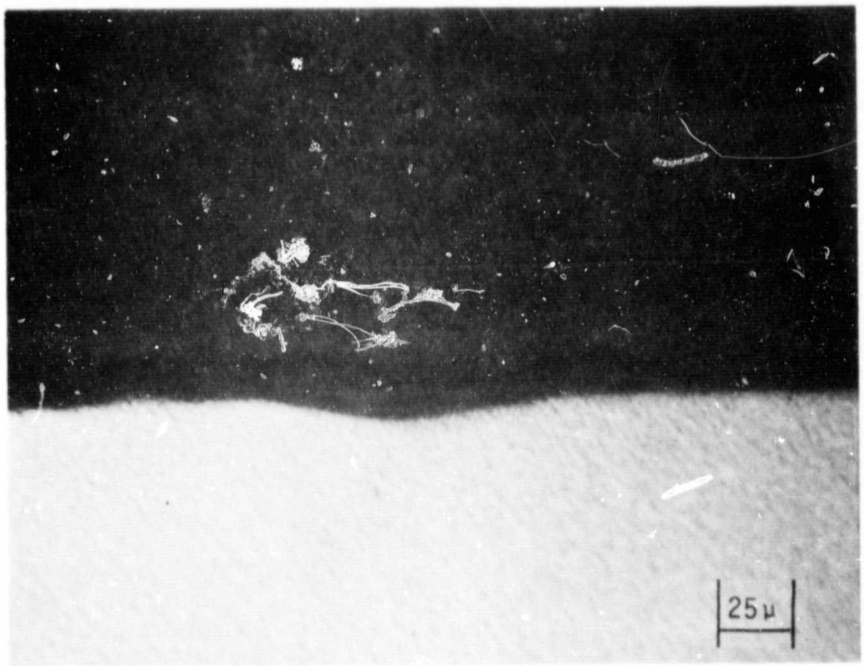


k) 7527, Liquid, ∿ 400X

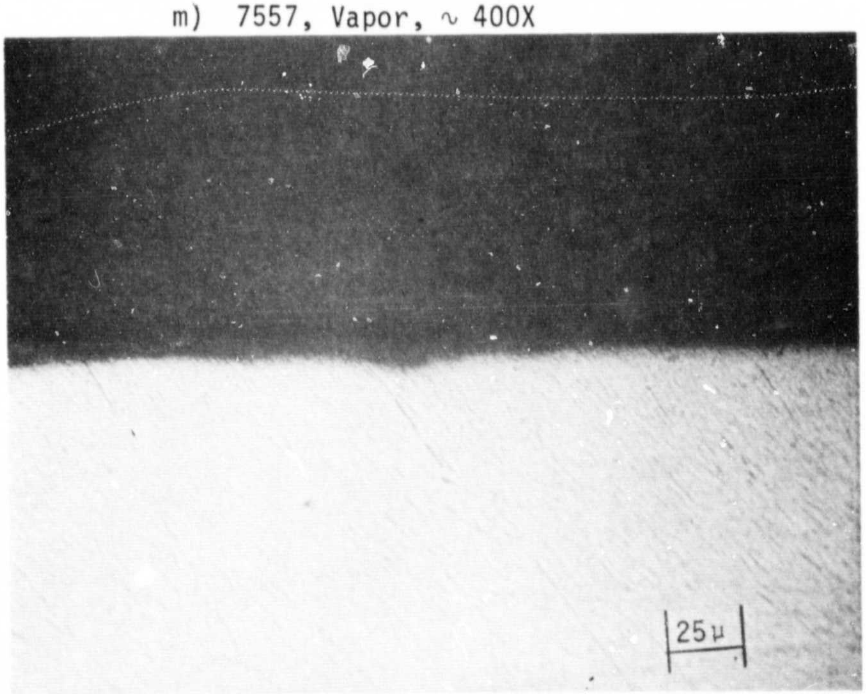


l) 7527, Liquid, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

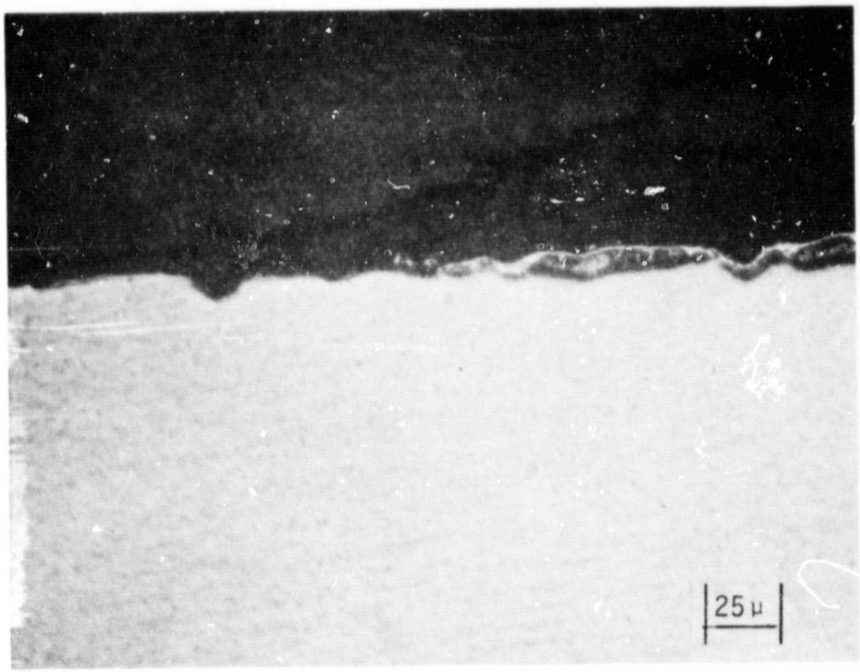


m) 7557, Vapor, ∿ 400X

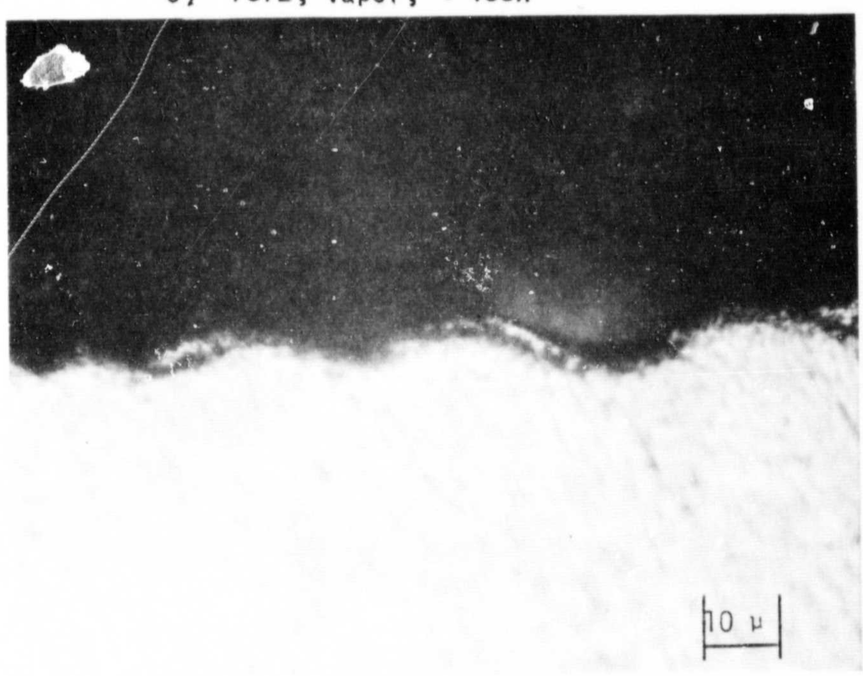


n) 7557, Liquid, ∿ 400X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens



o) 7572, Vapor, ∿ 400X



p) 7572, Vapor, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

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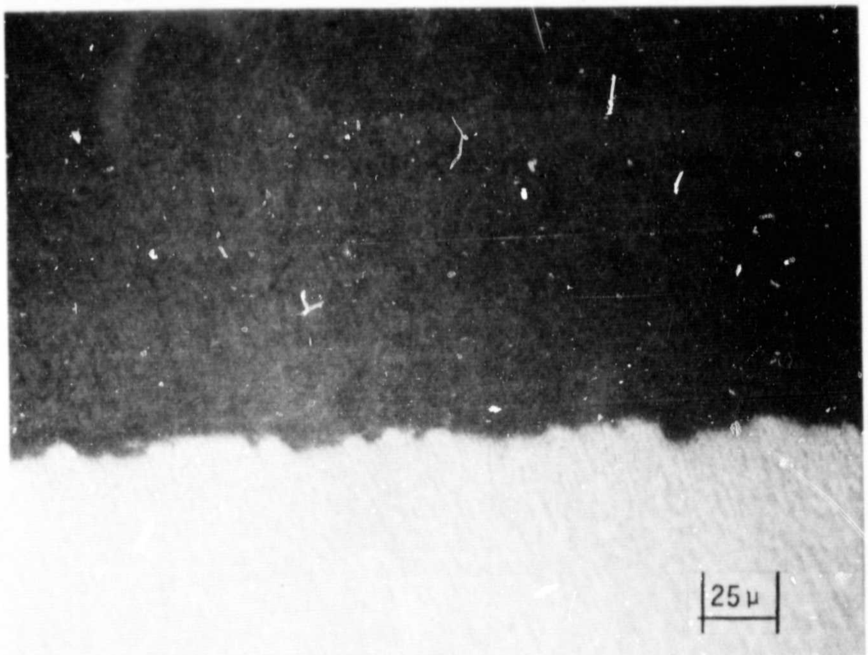
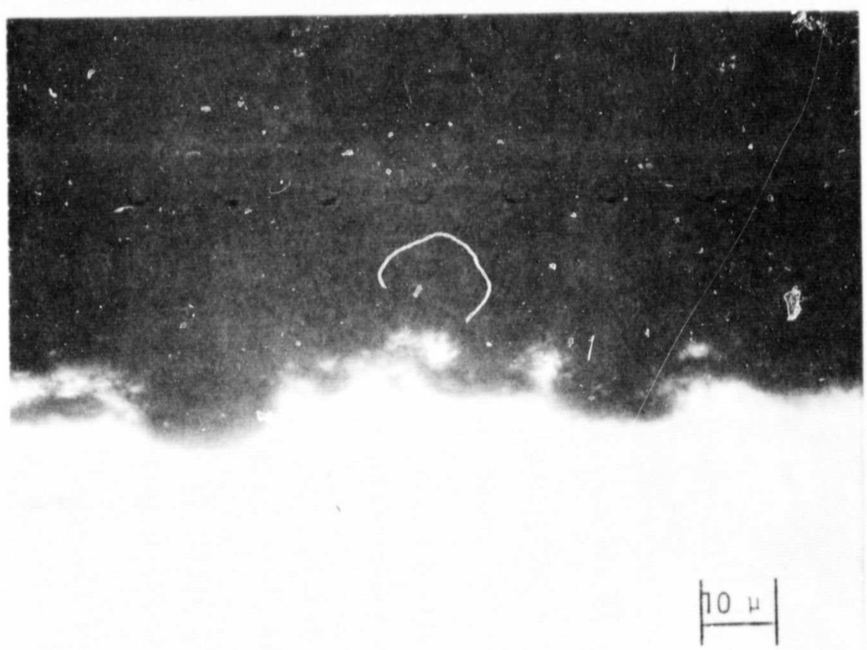
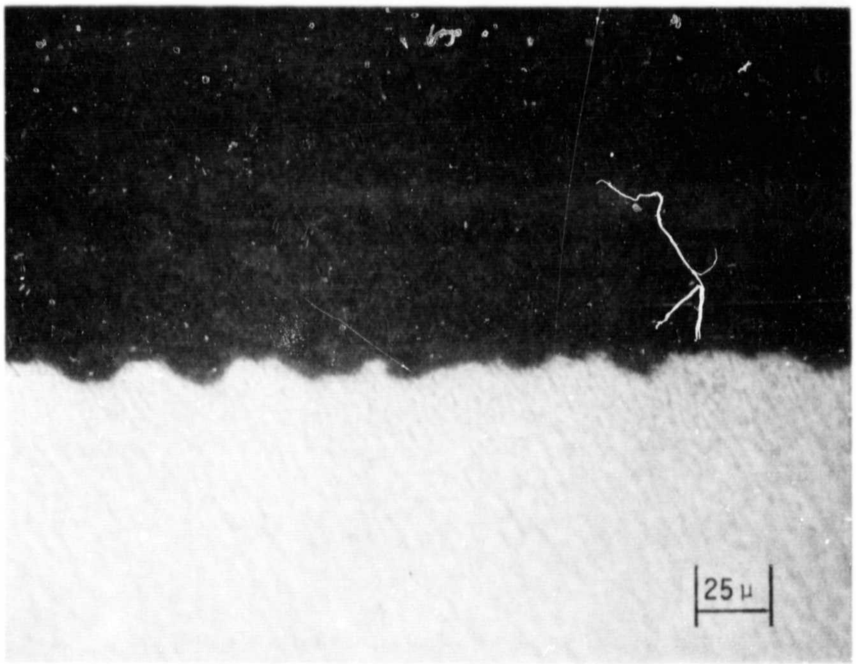


q) 7575, Vapor, ∿ 400X

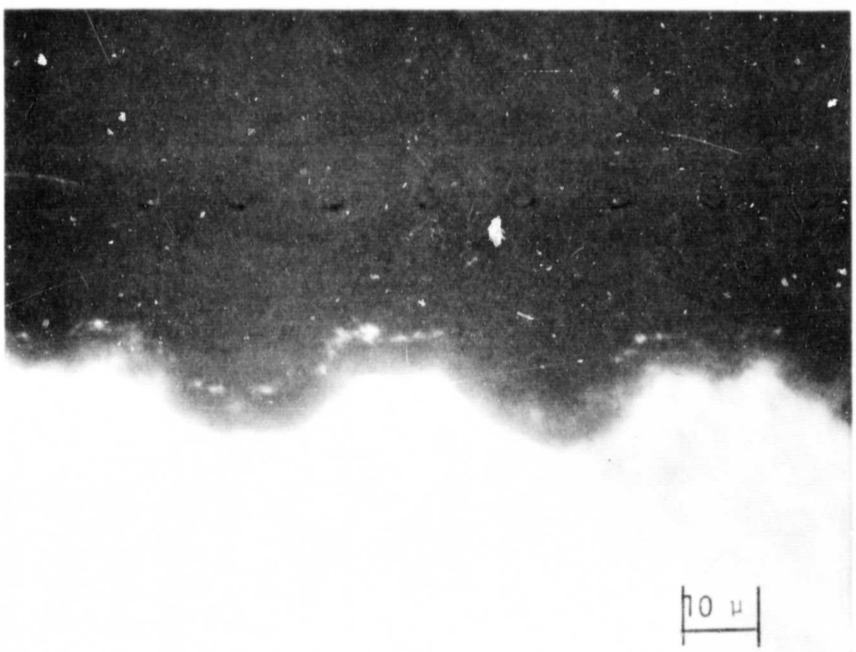


r) 7575, Vapor, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

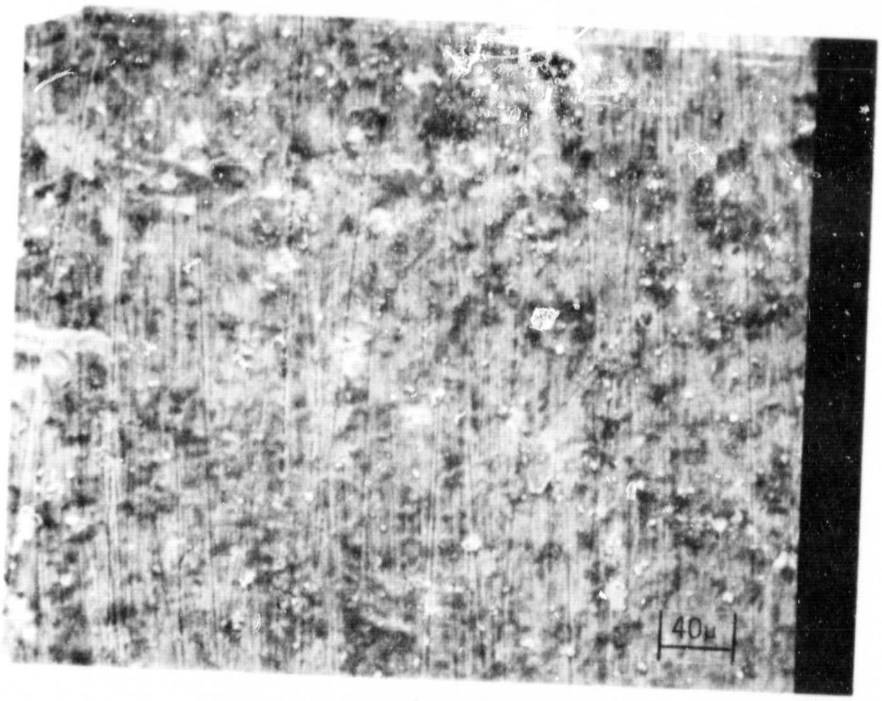


s) 7575, Liquid, ∿ 400X

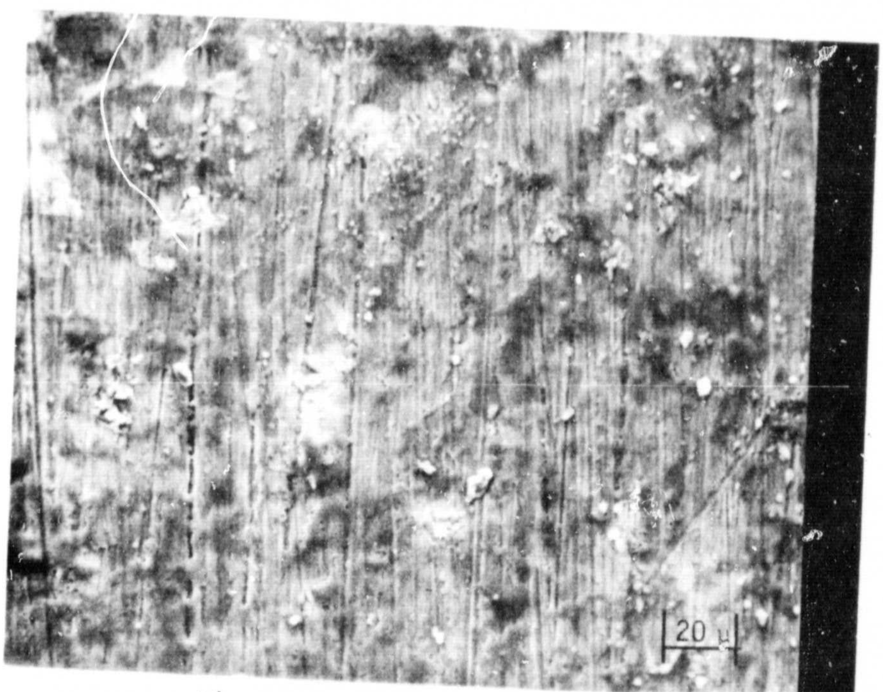


t) 7575, Liquid, ∿ 1000X

Figure F-3: Photomicrographs of Post-Immersion
(continued) 29-Month Specimens

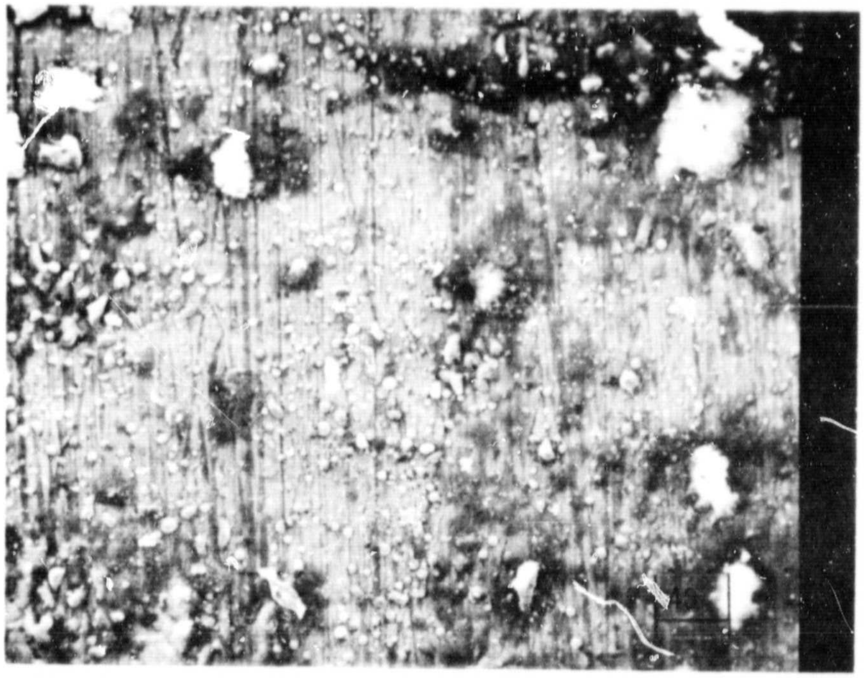


a) 7505, Vapor, ∿ 250X



b) 7505, Vapor, ∿ 500X

Figure F-4: SEM Photographs of Post-Immersion 29-Month Specimens

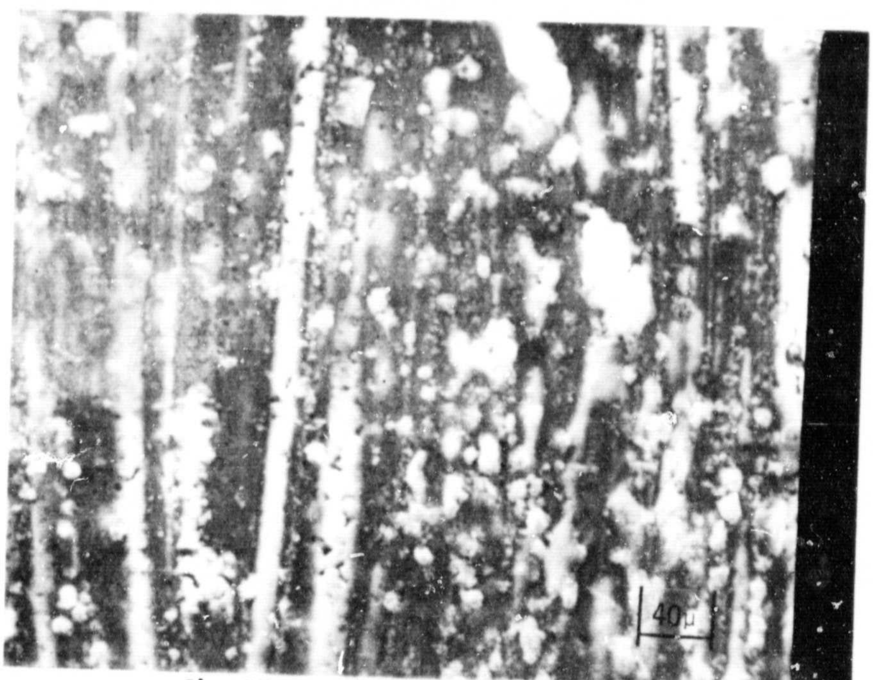


c) 7505, Liquid, ∿ 250X

d) 7505, Liquid, ∿ 500X

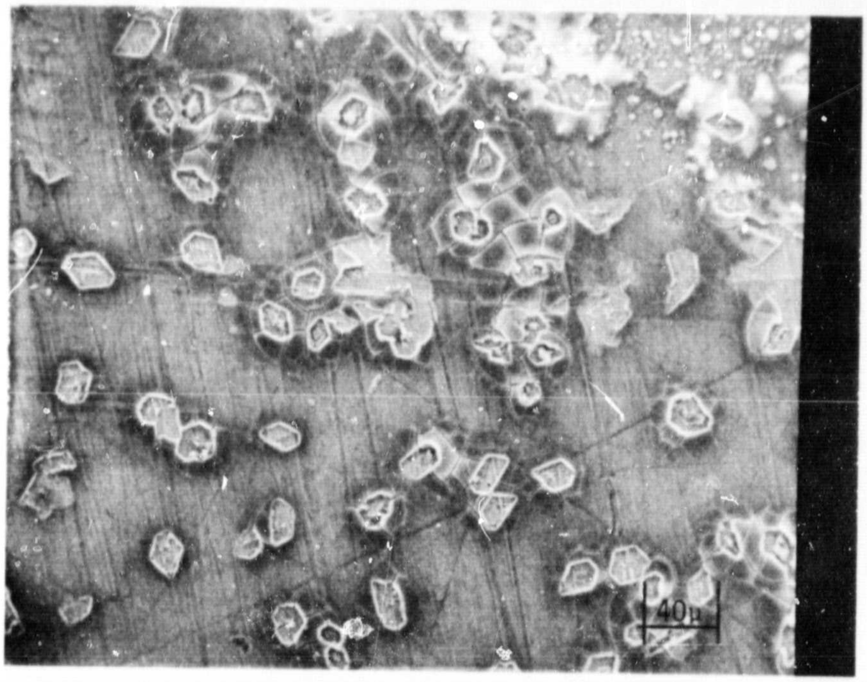
Figure F-4: SEM Photographs of Post-Immersion
(continued) 29-Month Specimens

e) 7527, Vapor, ~ 250X

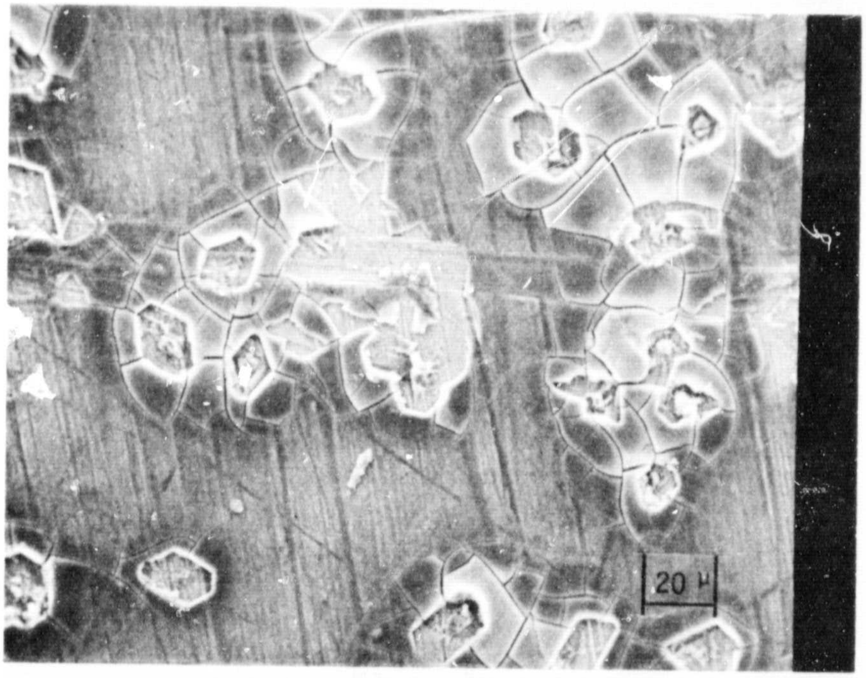


f) 7527, Liquid, ~ 250X

Figure F-4: SEM Photographs of Post-Immersion
(continued) 29-Month Specimens

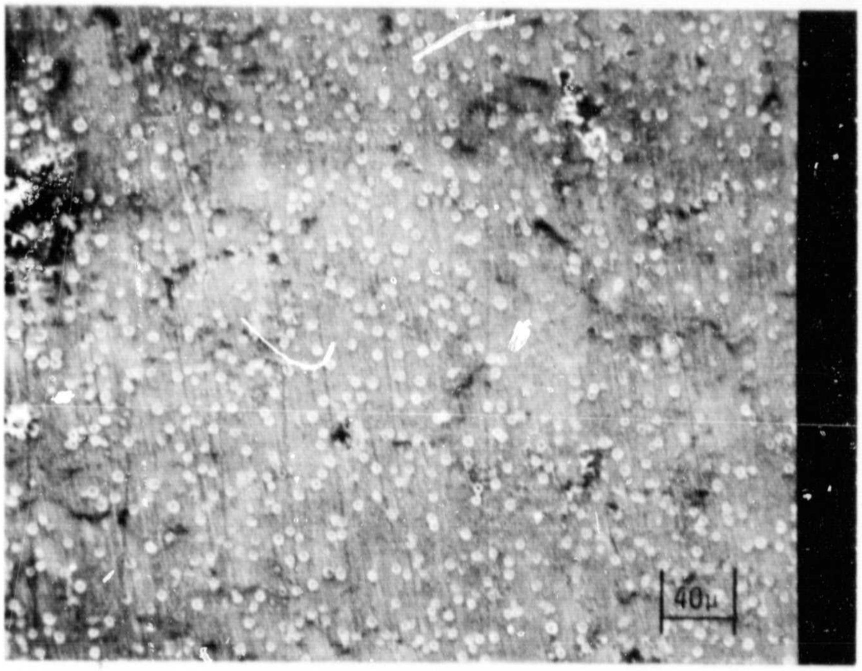


g) 7555, Vapor, ∿ 250X



h) 7555, Vapor, ∿ 500X

Figure F-4: SEM Photographs of Post-Immersion
(continued) 29-Month Specimens



i) 7555, Liquid, ∿ 250X

j) 7555, Liquid, ∿ 500X

Figure F-4: SEM Photographs of Post-Immersion
(continued) 29-Month Specimens

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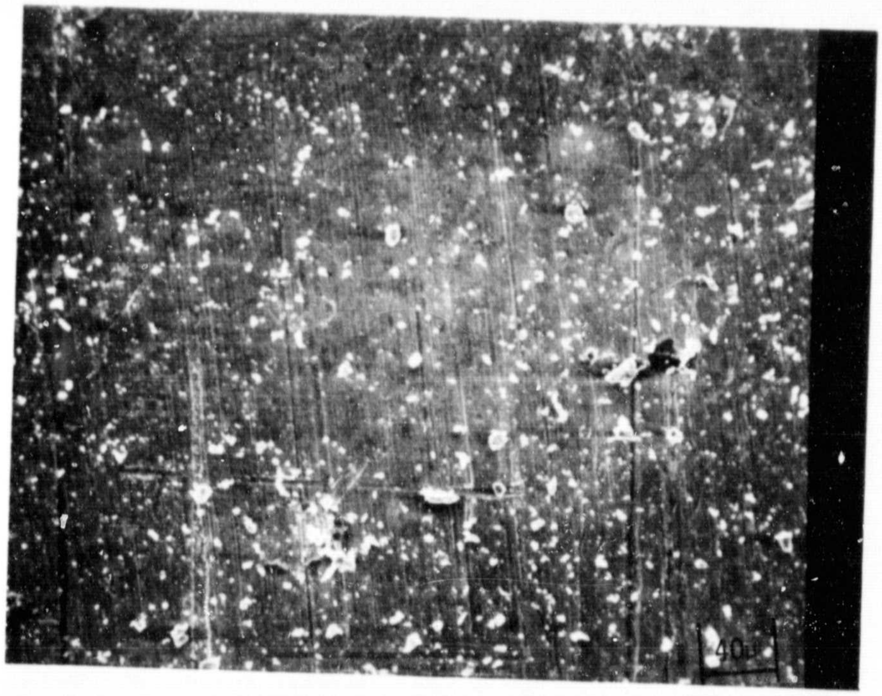
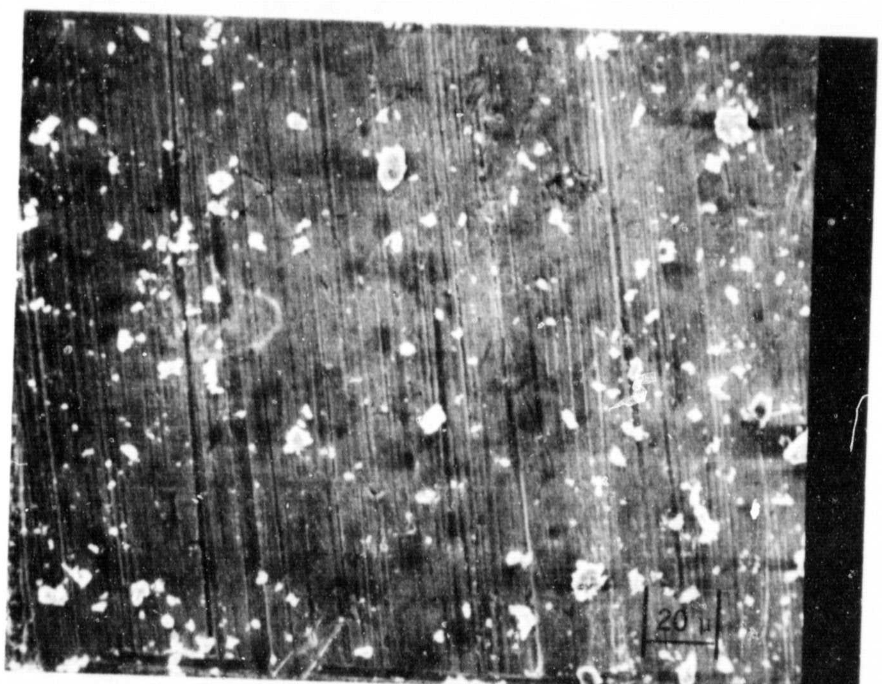
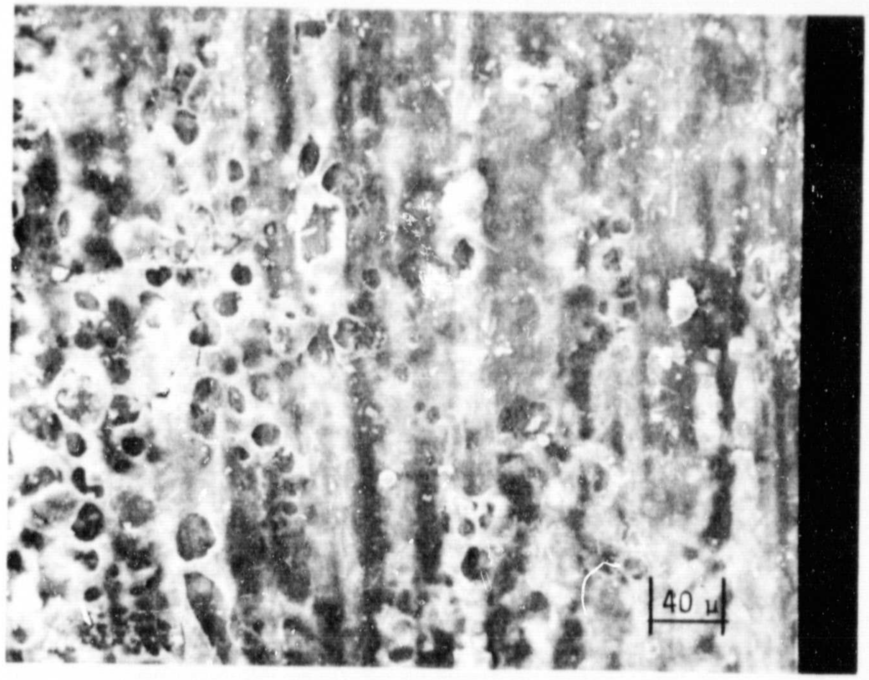


k) 7572, Vapor, ∿ 250X



l) 7572, Vapor, ∿ 500X

Figure F-4: SEM Photographs of Post-Immersion
(continued) 29-Month Specimens



m) 7572, Liquid, ∿ 250X

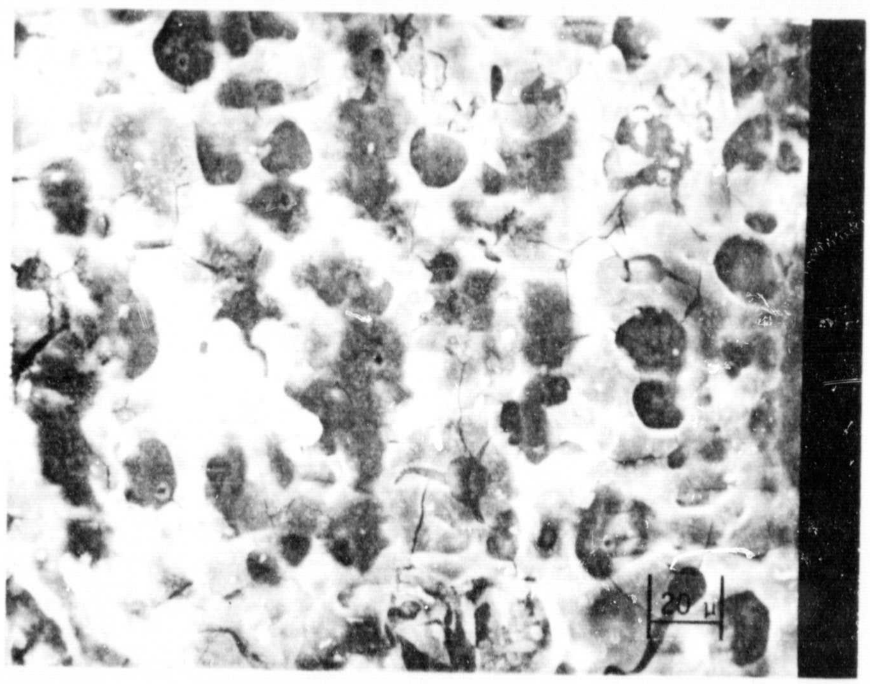


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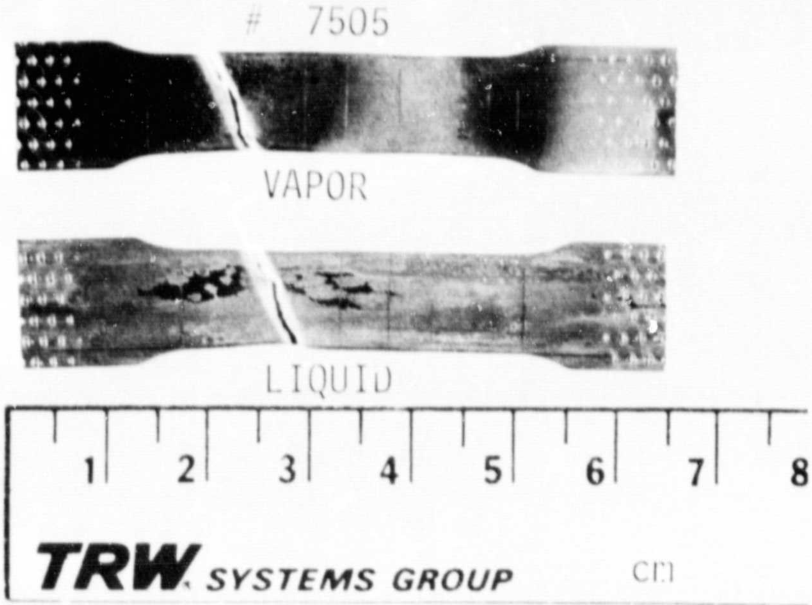
Figure F-4: SEM Photographs of Post-Immersion
(continued) 29-Month Specimens



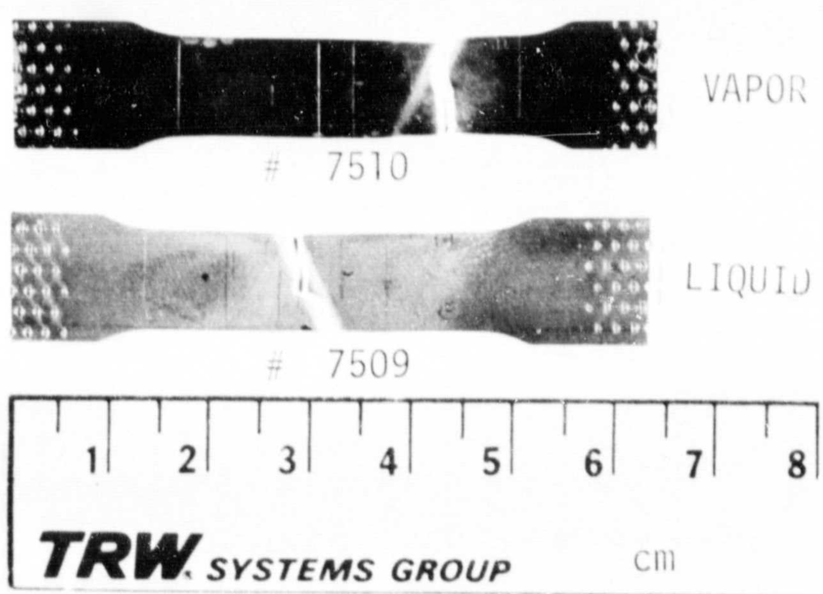
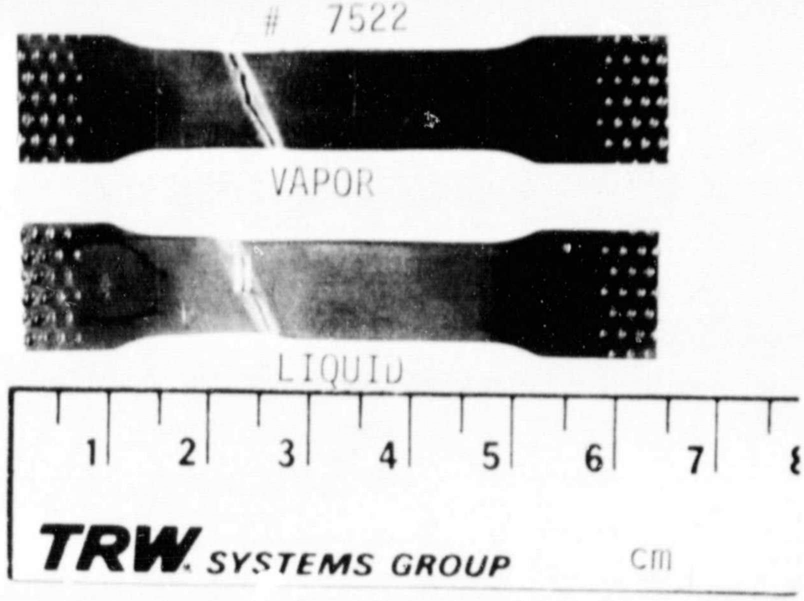


Figure F-5: Photographs of Fractured Tensile Coupons, Post-Immersion 29-Month Tests, 298 K



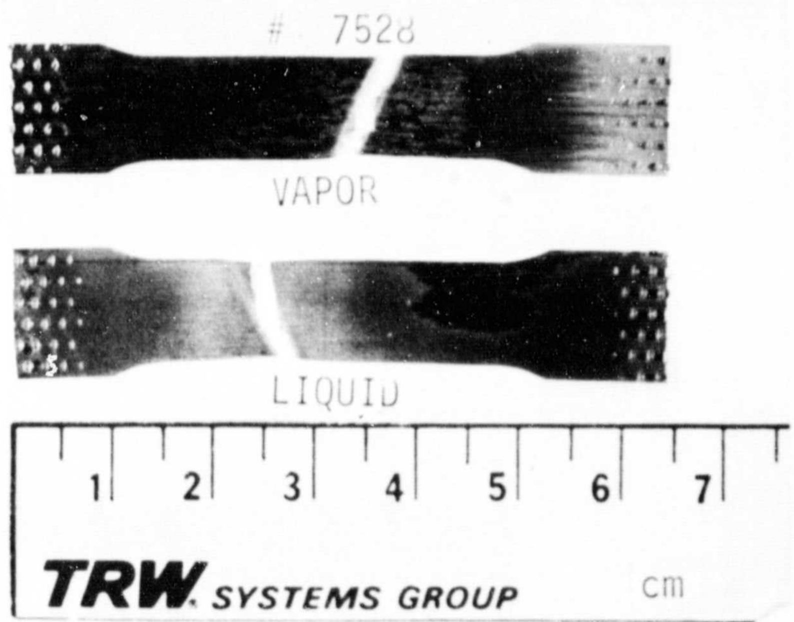


Figure F-5: Photographs of Fractured Tensile Coupons,
(continued) Post-Immersion 29-Month Tests, 298 K.

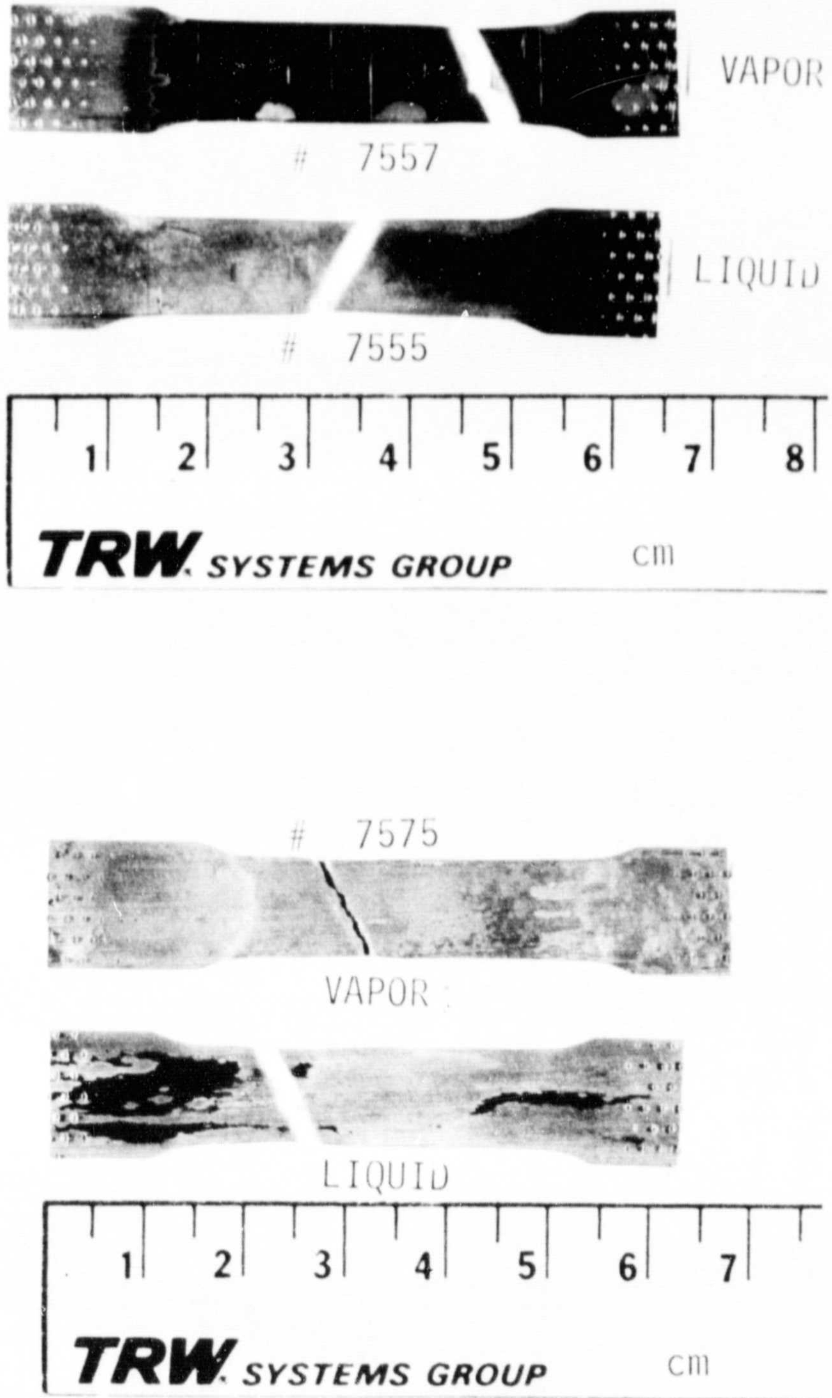
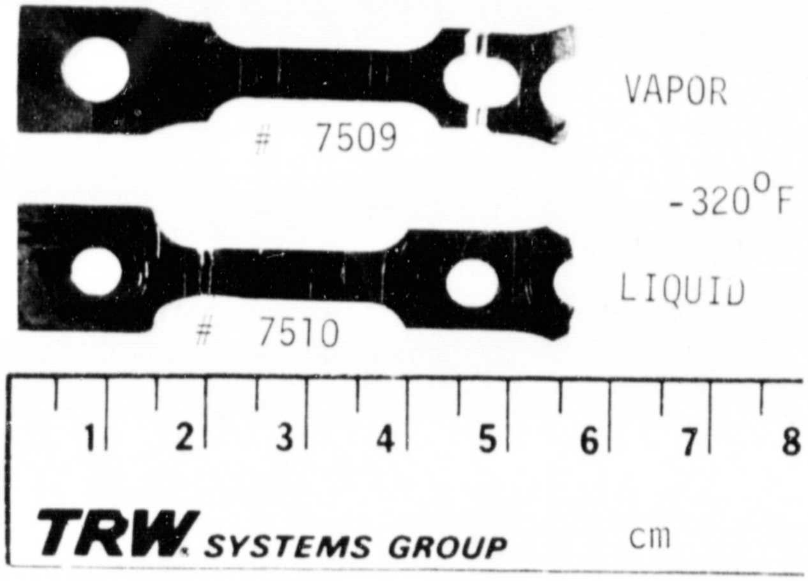


Figure F-5: Photographs of Fractured Tensile Coupons,
(continued) Post-Immersion 29-Month Tests, 298 K.



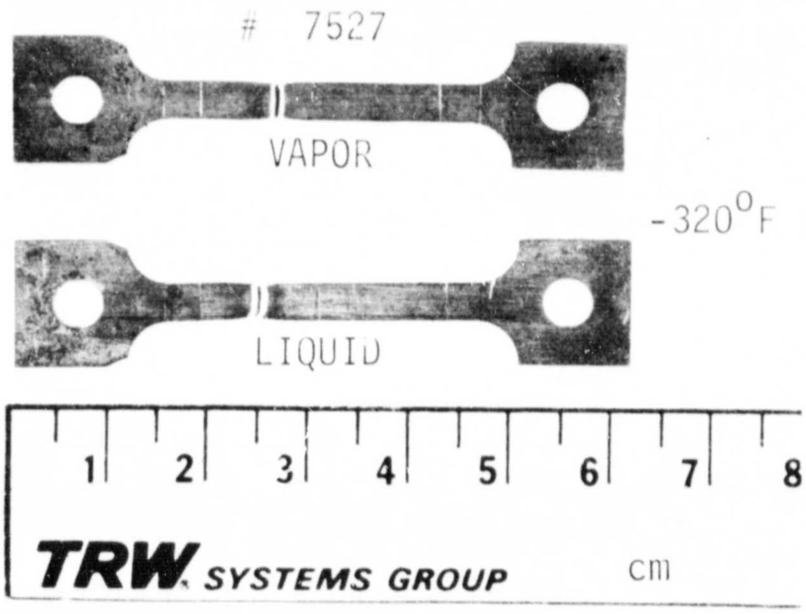


Figure F-6: Photographs of Fractured Tensile Coupons Post-Immersion 29-Month Tests, 77 K

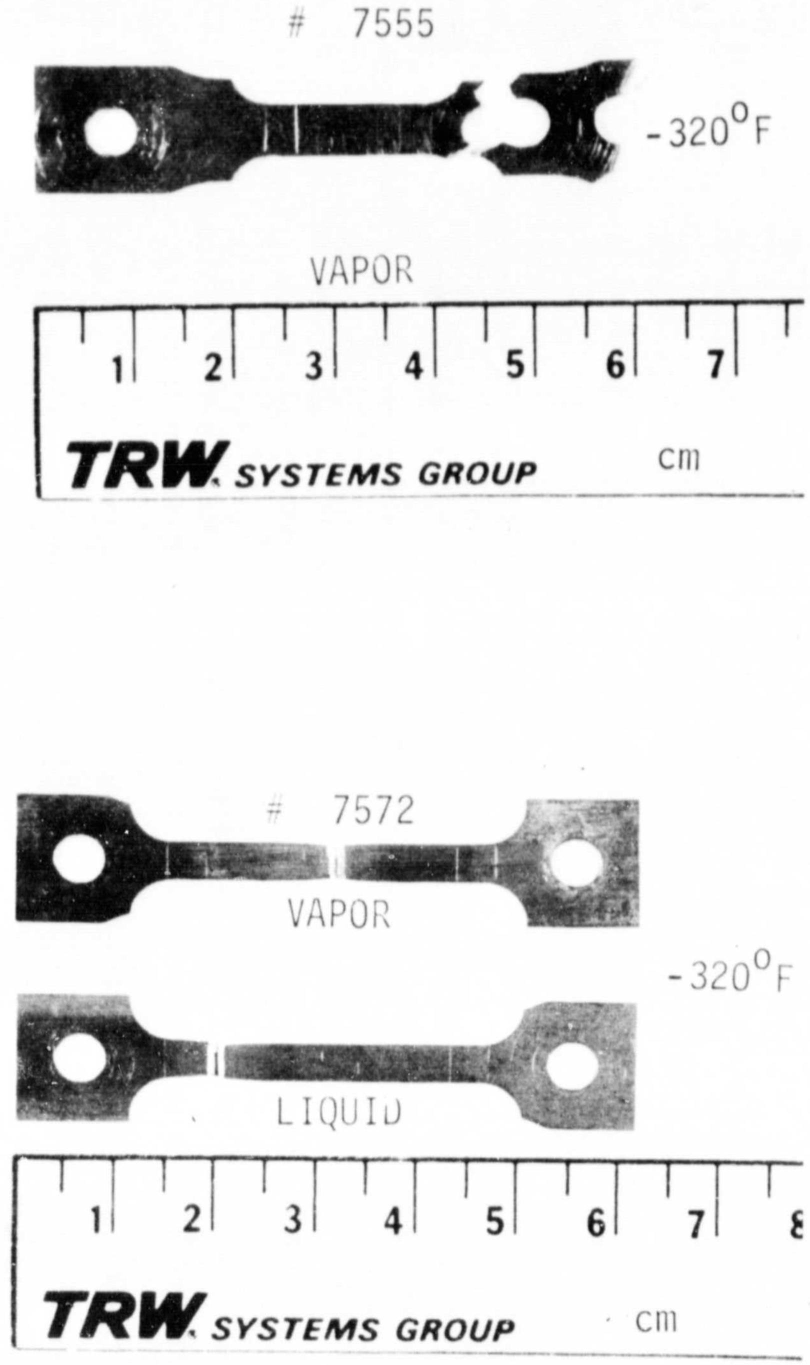


Figure F-6: Photographs of Fractured Tensile Coupons,
(continued) Post-Immersion 29-Month Tests, 77 K

TABLE F-I: PIT DEPTH FREQUENCY DISTRIBUTION: 29-MONTH IMMERSION

Specimen No.	Percent of Pits in Various Depth Ranges (10^{-4}cm)											
	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10	Group 11	Group 12
Range Mean	0.01-0.64 (0.325)	0.65-1.28 (0.965)	1.29-1.92 (1.61)	1.93-2.56 (2.24)	2.57-3.20 (2.89)	3.21-3.84 (3.53)	3.85-4.48 (4.17)	4.49-5.12 (4.81)	5.13-5.76 (5.61)	5.77-6.40 (6.09)	6.41-7.04 (6.73)	>7.04
7505 Liquid	0	0	41.7	16.7	16.7	0	8.3	16.7	0	0	0	0
7505 Vapor	0	0	40.0	60.0	0	0	0	0	0	0	0	0
7510 Liquid	0	36.4	36.4	27.2	0	0	0	0	0	0	0	0
7510 Vapor	0	0	67.7	11.1	11.1	0	0	0	11.1	0	0	0
7527 Liquid	0	0	0	9.1	0	8.2	0	27.3	9.1	9.1	0	27.3
7527 Vapor	0	0	23.5	11.8	17.6	5.9	0	5.9	0	23.5	5.9	5.9
7557 Liquid	0	8.3	66.7	25.0	0	0	0	0	0	0	0	0
7557 Vapor	14.3	0	57.1	14.3	0	14.3	0	0	0	0	0	0
7572 Vapor	0	0	0	37.5	0	12.5	0	6.3	0	25.0	6.3	12.5
7575 Liquid	0	0	10.5	21.1	21.1	15.8	5.3	5.3	5.3	0	0	15.8
7575 Vapor	0	0	2.9	0	5.9	11.8	5.9	17.6	11.8	2.9	8.8	32.4

TABLE F-II: PIT AREA FREQUENCY DISTRIBUTION: 29-MONTH IMMERSION COUPONS

Specimen No.	Percent of Pits in Various Area Ranges - 10^{-7} cm^2 (Average)							
Range Mean	Group 1 0.001-0.049 (0.025)	Group 2 0.050-0.009 (0.0745)	Group 3 0.100-0.490 (0.295)	Group 4 0.500-0.990 (0.0745)	Group 5 1.000-4.999 (3.000)	Group 6 5.000-9.999 (7.500)	Group 7 10.000-14.999 (12.000)	Group 8 15.000-19.999 (17.500)
7505 Liquid	0	0	8.3	0	41.7	33.3	8.3	8.3
7505 Vapor	0	0	20.0	0	80.0	0	0	0
7510 Liquid	0	0	9.1	0	72.7	18.2	0	0
7510 Vapor	0	0	0	11.1	44.4	33.3	11.1	0
7527 Liquid	0	0	0	0	9.1	0	9.1	9.1
7527 Vapor	0	0	0	5.9	29.4	5.9	11.8	0
7557 Liquid	0	0	16.7	0	50.0	16.7	8.3	0
7557 Vapor	0	0	57.1	0	42.9	0	0	0
7572 Vapor	0	0	0	0	18.8	6.3	12.5	18.8
7575 Liquid	0	0	0	0	52.6	31.6	10.5	5.3
7575 Vapor	0	0	2.9	0	41.2	23.5	8.8	0

	Group 9 20.000-24.999 (22.500)	Group 10 25.000-29.999 (27.500)	Group 11 30.000-34.999 (32.500)	Group 12 35.000-39.999 (37.500)	Group 13 40.000-44.999 (42.500)	Group 14 45.000-49.999 (47.500)	Group 15 50.000-54.999 (52.500)	Group 16 >52.500
7505 Liquid	0	0	0	0	0	0	0	0
7505 Vapor	0	0	0	0	0	0	0	0
7510 Liquid	0	0	0	0	0	0	0	0
7510 Vapor	0	0	0	0	0	0	0	0
7527 Liquid	9.1	18.2	0	0	9.1	0	27.3	9.1
7527 Vapor	17.6	5.9	5.9	5.9	0	5.9	0	5.9
7557 Liquid	0	0	0	0	0	0	0	8.3
7557 Vapor	0	0	0	0	0	0	0	0
7572 Vapor	6.3	12.5	12.5	6.3	0	6.3	0	0
7575 Liquid	0	0	0	0	0	0	0	0
7575 Vapor	5.9	8.8	0	0	0	0	5.9	2.9

TABLE F-III. PIT DEPTH/DIAMETER RATIO FREQUENCY DISTRIBUTION: 29-MONTH IMMERSION COUPONS

Specimen No.	Percent of Pits with Various Depth/Diameter (1/d) Ratios							
	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
Ratio Range Mean	0.01-0.20 (0.105)	0.21-0.41 (0.31)	0.42-0.62 (0.52)	0.63-0.83 (0.73)	0.84-1.04 (0.94)	1.05-1.25 (1.15)	1.26-2.00 (1.63)	>2.00
7505 Liquid	16.7	58.3	16.7	8.3	0	0	0	0
7505 Vapor	0	40.0	20.0	40.0	0	0	0	0
7510 Liquid	44.4	44.4	11.1	0	0	0	0	0
7510 Vapor	45.5	45.5	9.0	0	0	0	[illegible]	0
7527 Liquid	17.6	64.7	17.6	0	0	0	0	0
7527 Vapor	18.2	54.5	18.2	9.1	0	0	0	0
7557 Liquid	41.7	41.7	16.7	0	0	0	0	0
7557 Vapor	0	42.9	14.3	28.6	14.3	0	0	0
7572 Vapor	6.3	75.0	6.3	0	6.3	6.3	0	0
7575 Liquid	5.3	31.6	31.6	15.8	5.3	5.3	0	5.3
7575 Vapor	0	26.5	35.3	8.8	5.9	8.8	11.8	2.9

APPENDIX G

APPEARANCE OF 39-MONTH IMMERSION COUPONS

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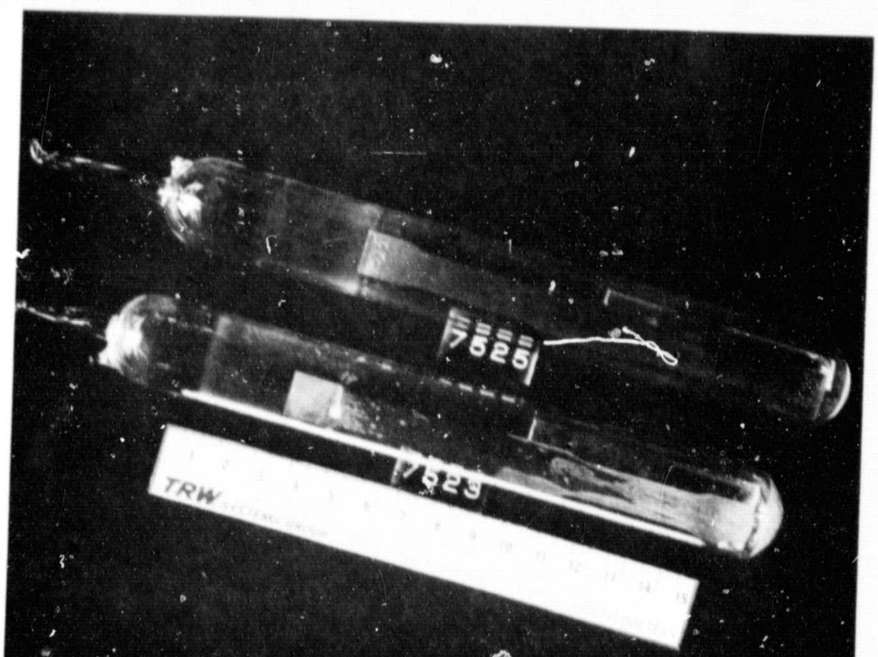
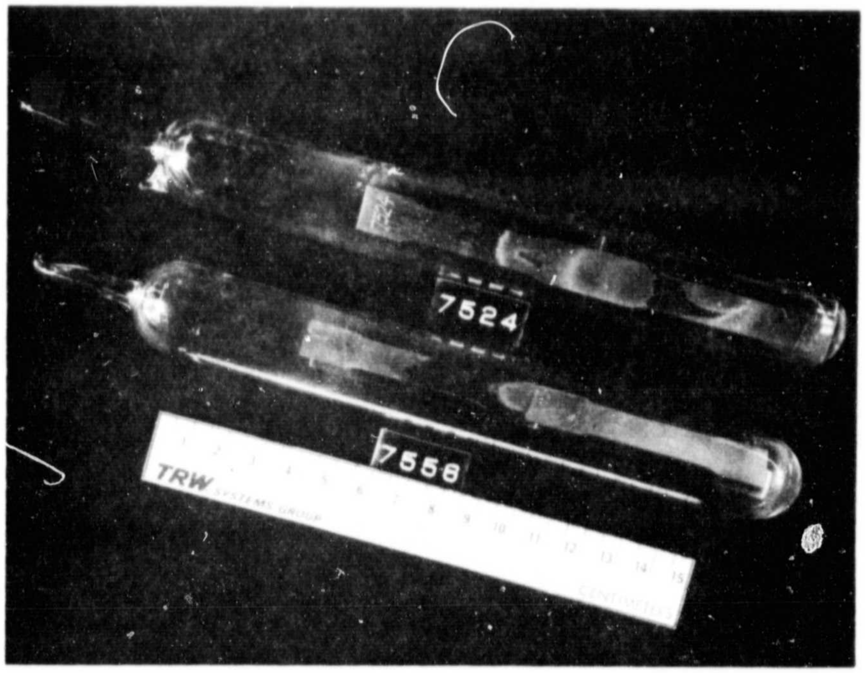
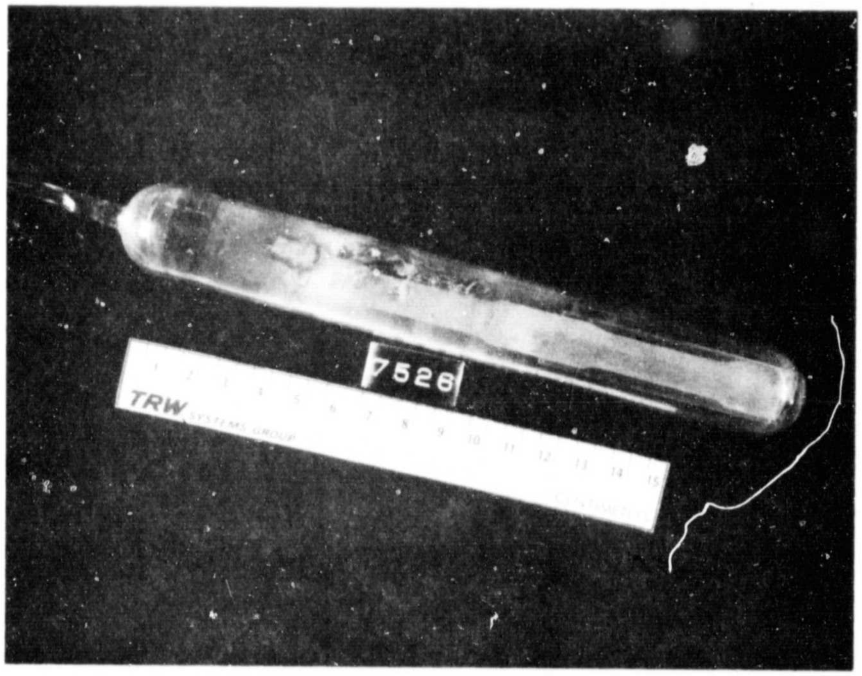


#7523 has high SiF_4 content

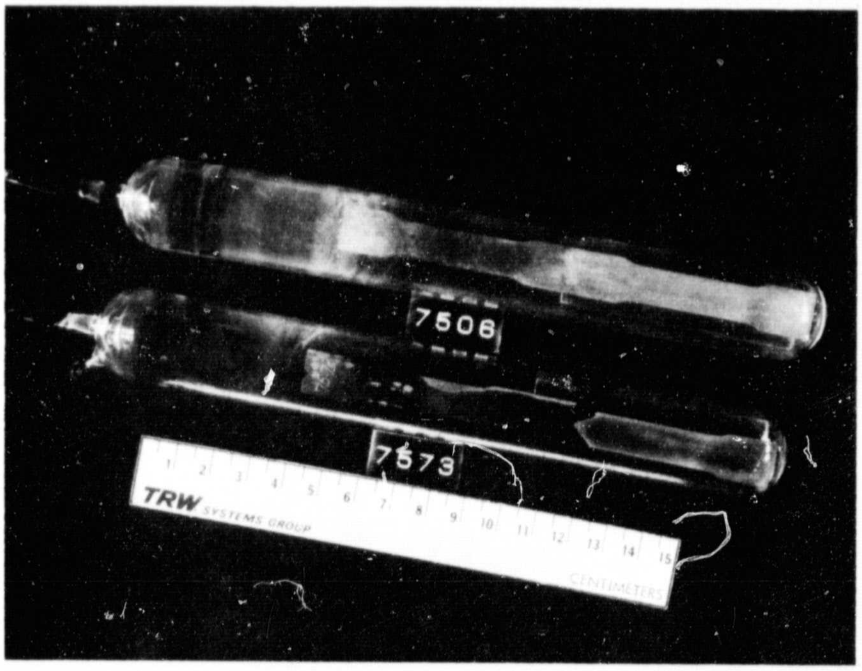
Figure G-1. Double-Dogbone Specimens in Capsules After 39-Month GF_2/LF_2 Immersion





#7526 had dark F_2, high SiF_4, and Ti salts

Figure G-1. Double-Dogbone Specimens in Capsules After 39-Month GF_2/LF_2 Immersion (Continued)



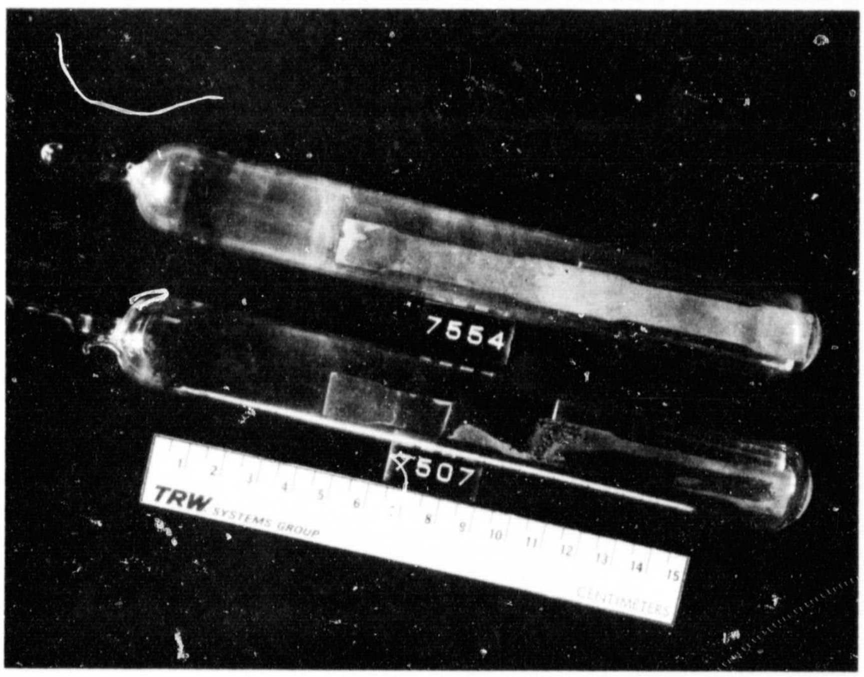


Figure G-1. Double-Dogbone Specimens in Capsules After 39-Month GF_2/LF_2 Immersion (Continued)

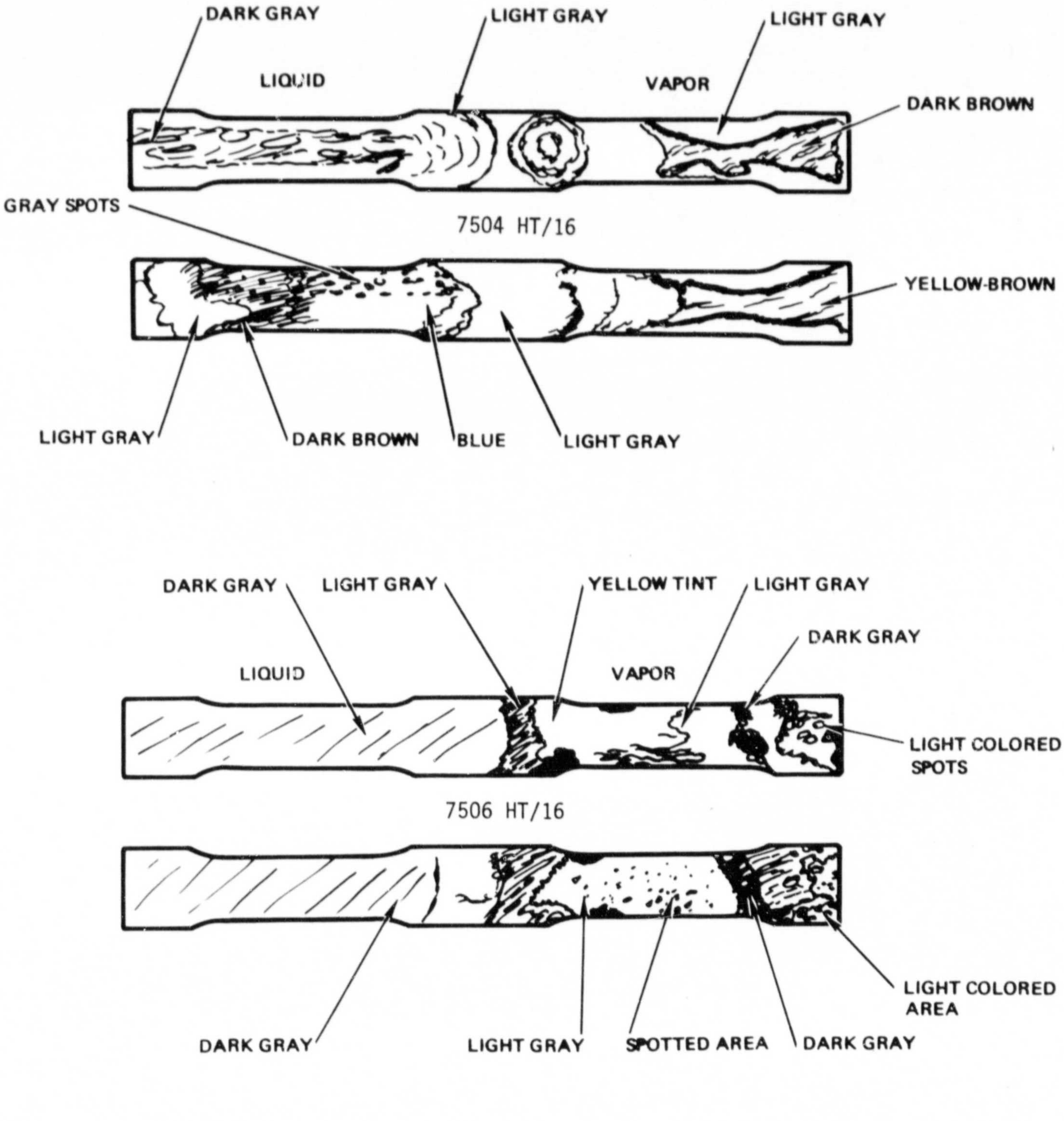
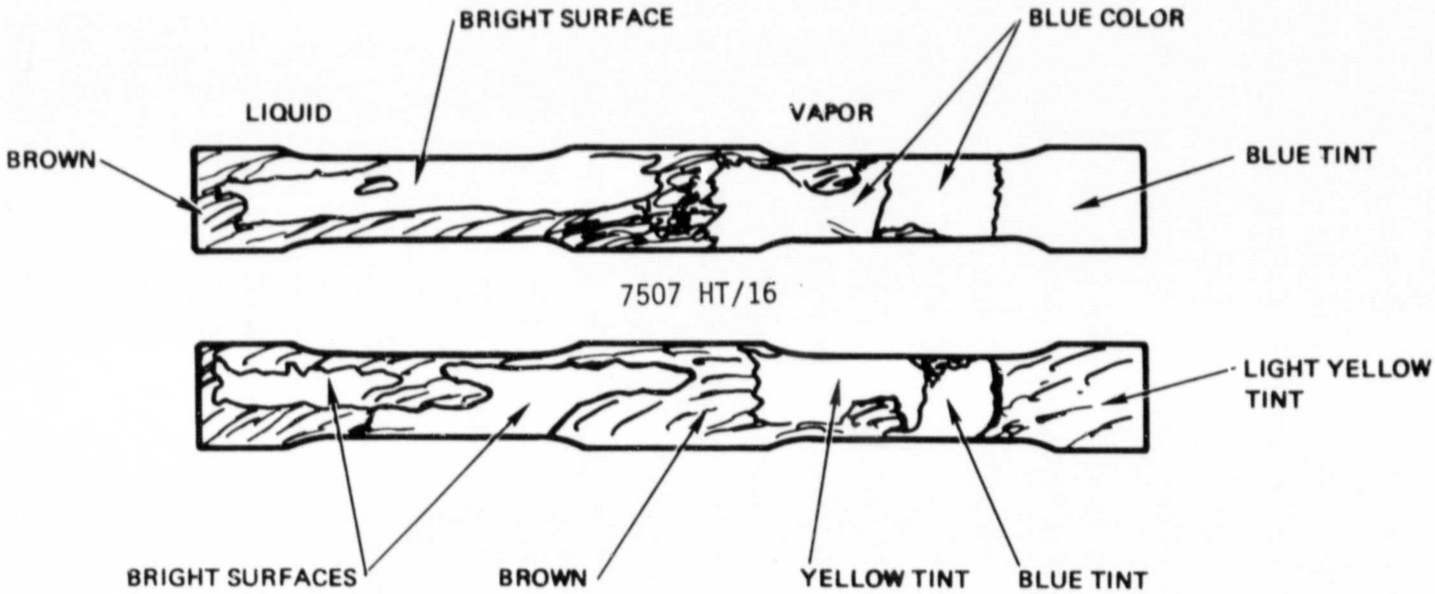
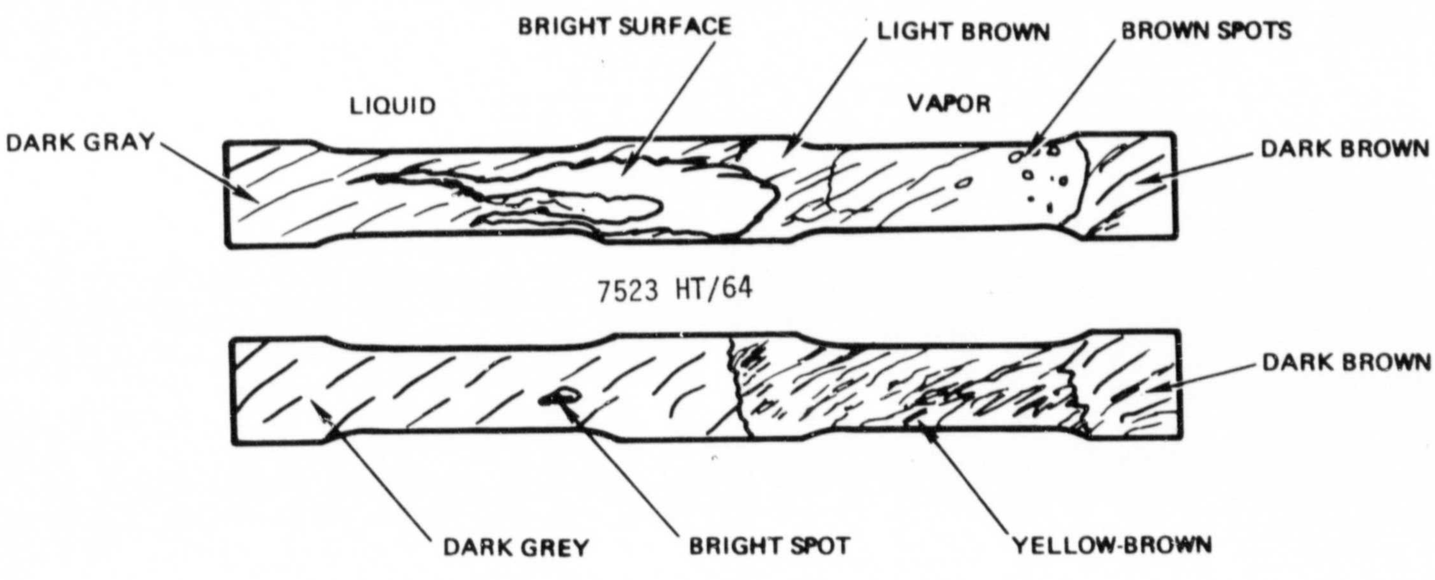


Figure G-2. Maps of Surfaces of 39-Month Specimens



7507 HT/16



7523 HT/64

Figure G-2. Maps of Surfaces of 39-Month Specimens (Continued)

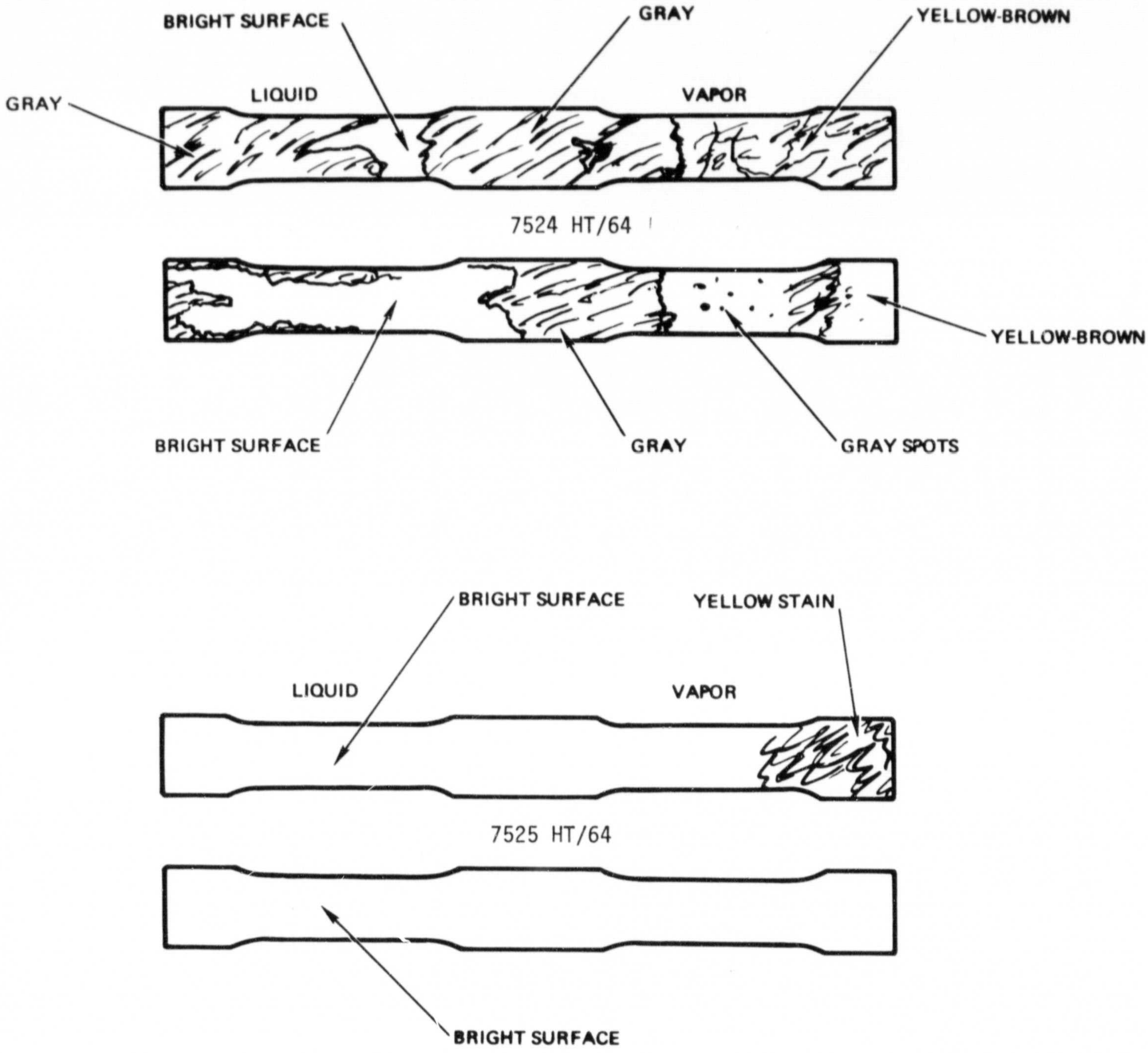
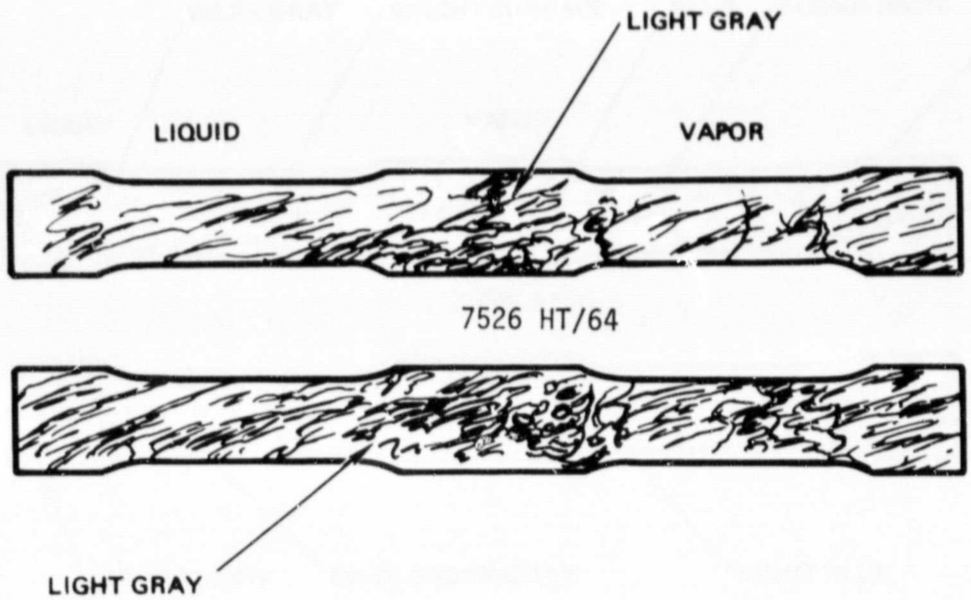


Figure G-2. Maps of Surfaces of 39-Month Specimens (Continued)



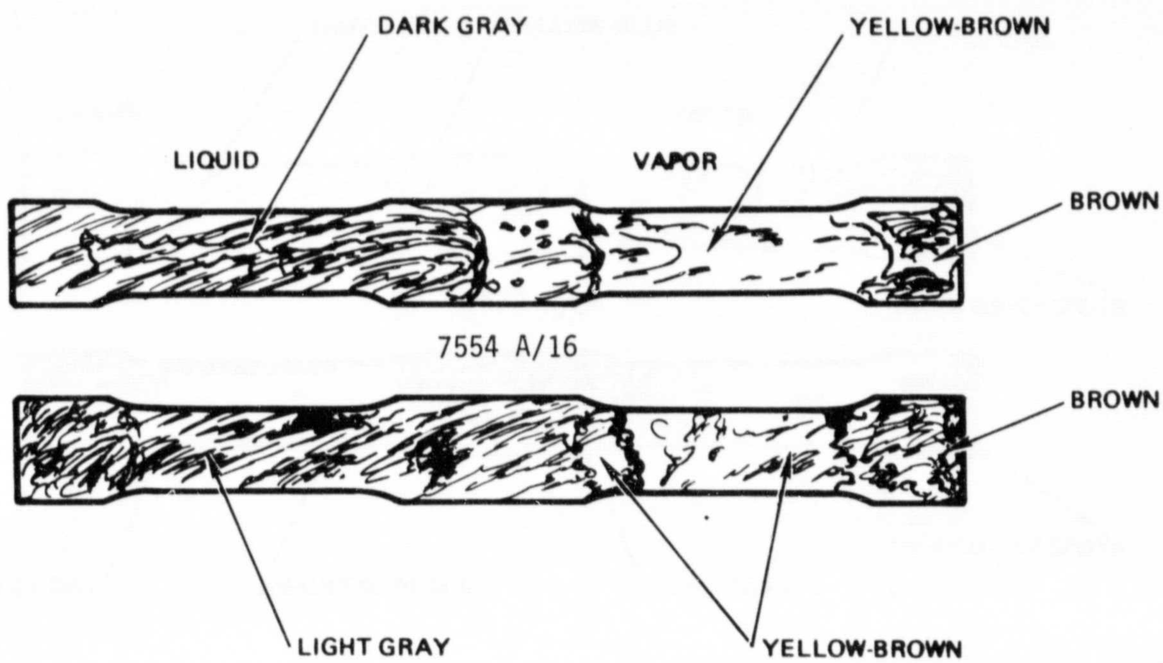
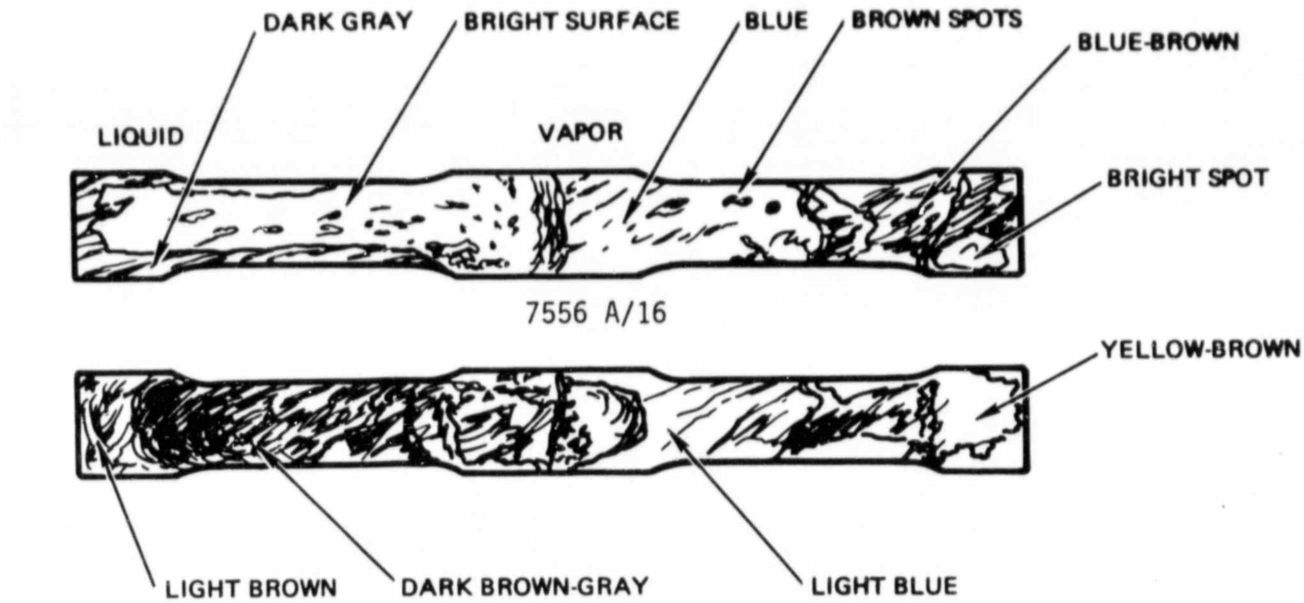


Figure G-2. Map of Surfaces of 39-Month Specimens (Continued)



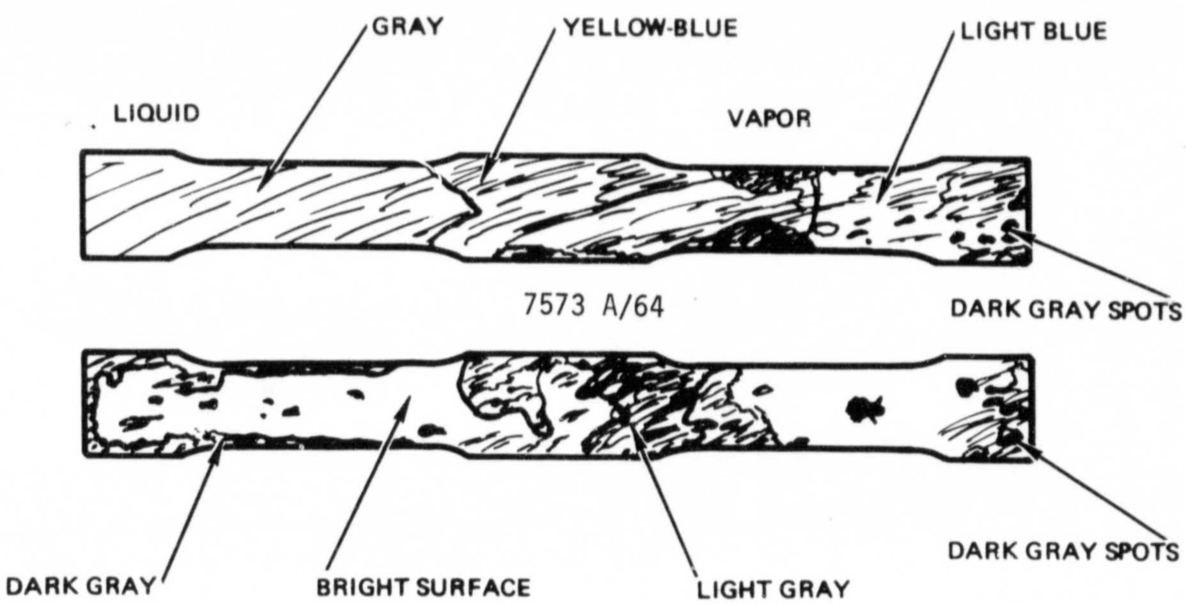


Figure G-2. Map of Surfaces of 39-Month Specimens (Continued)

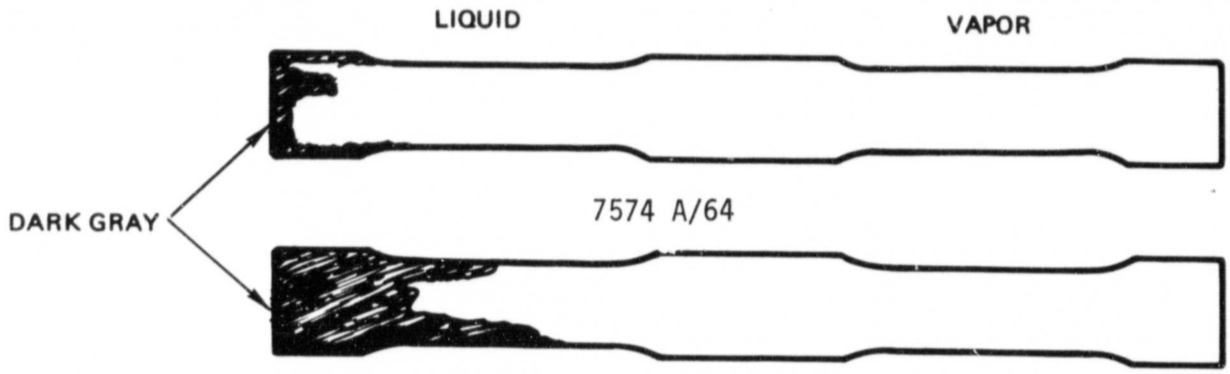


Figure G-2. Map of Surfaces of 39-Month Specimens (Continued)

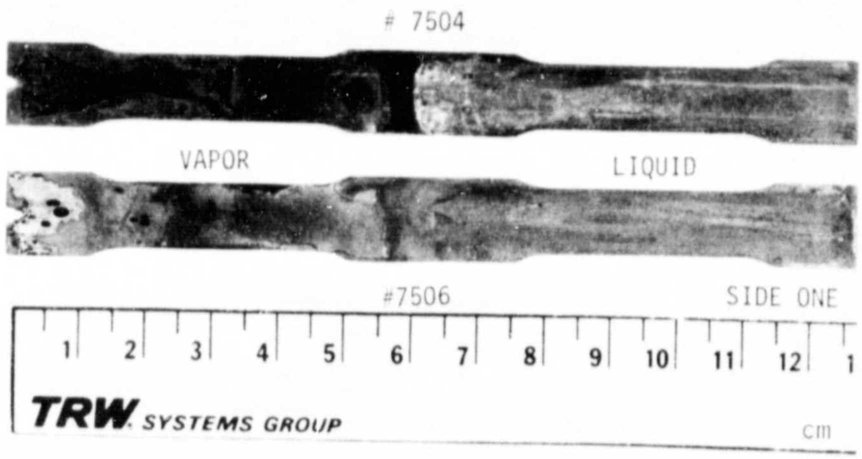
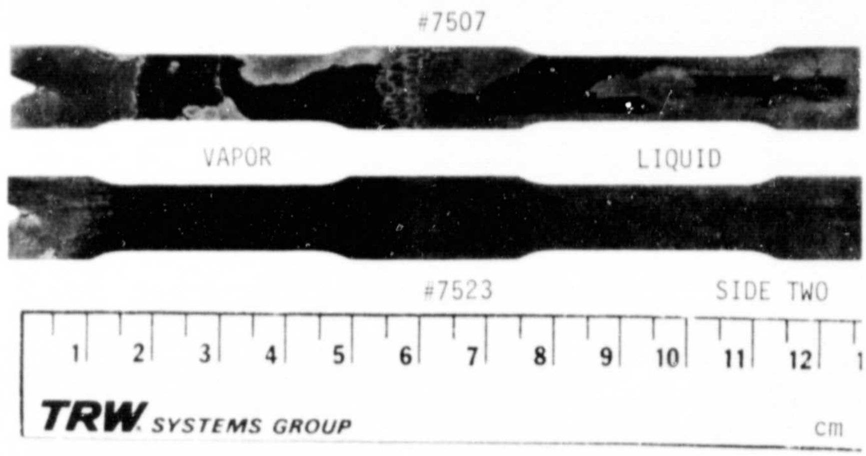


Figure G-3. Specimen Appearances After 39-Month F2 Immersion



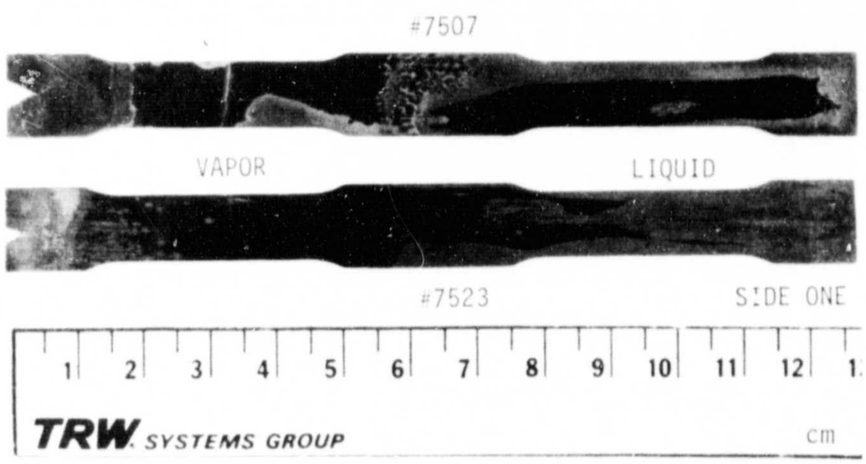
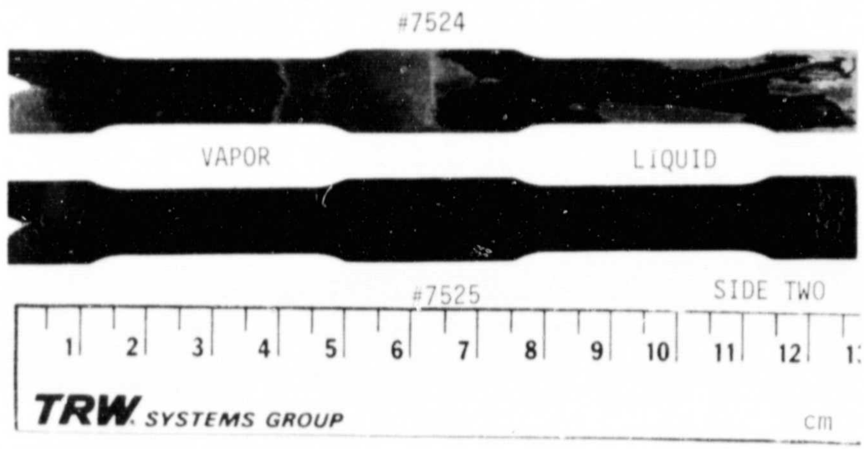


Figure G-3. Specimen Appearances After 39-Month F2 Immersion (Continued)



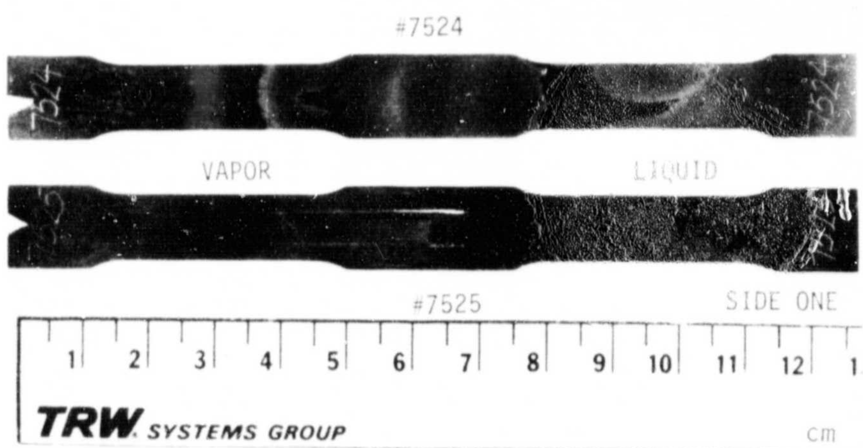
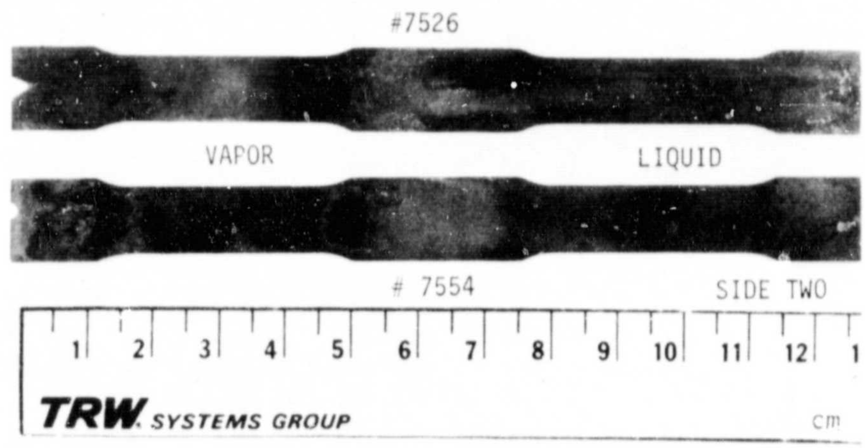


Figure G-3. Specimen Appearances After 39-Month F2 Immersion (Continued)



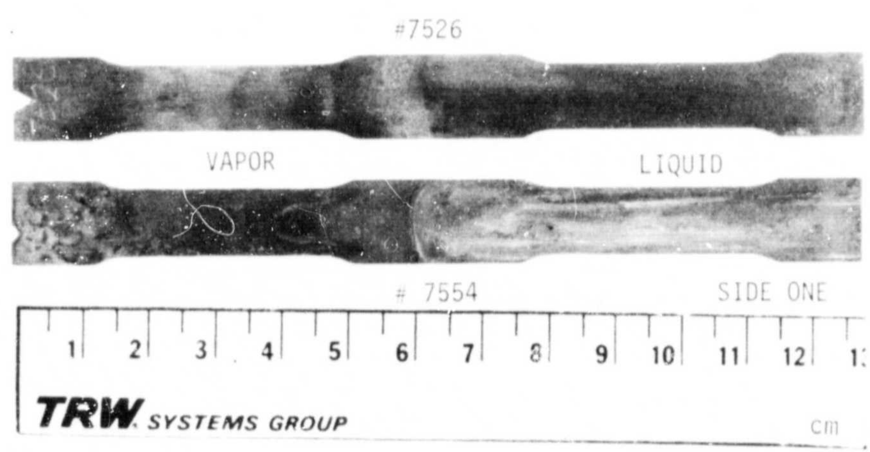
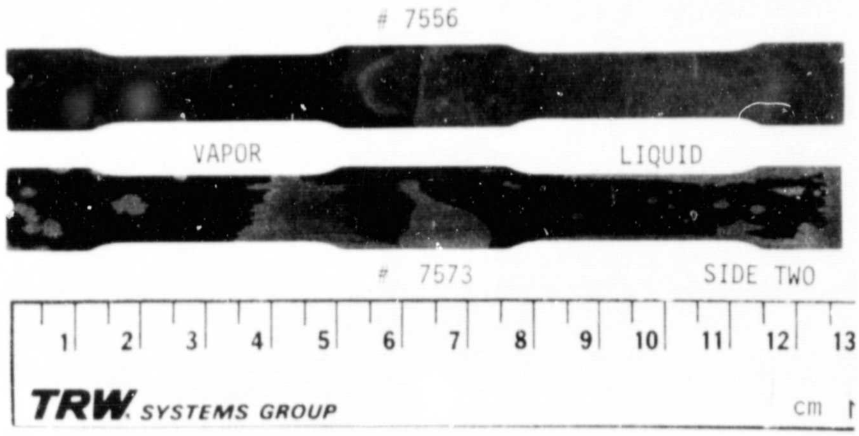


Figure G-3. Specimen Appearances After 39-Month F2 Immersion (Continued)



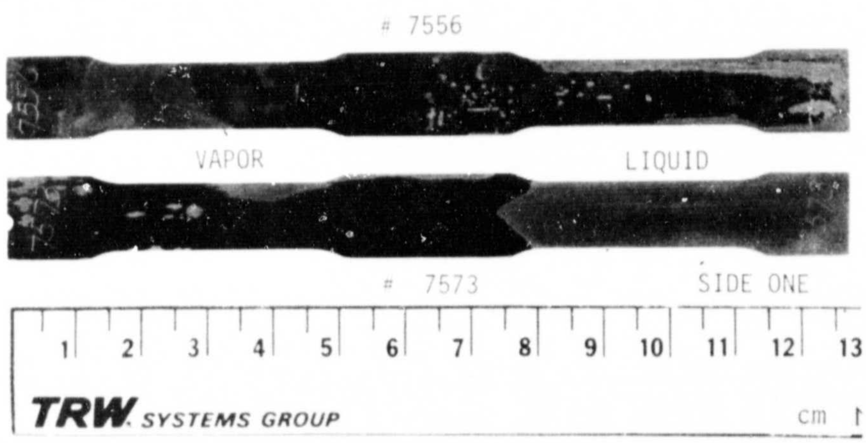
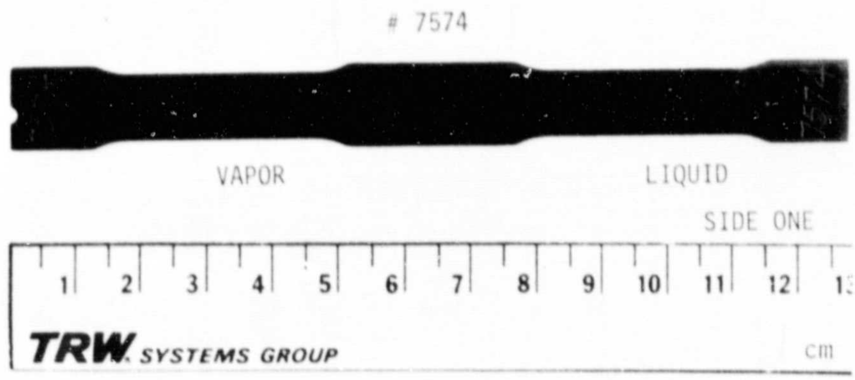


Figure G-3. Specimen Appearances After 39-Month F2 Immersion (Continued)



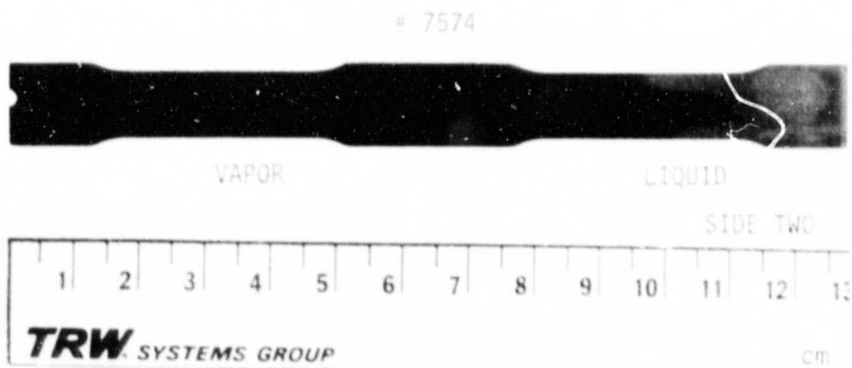
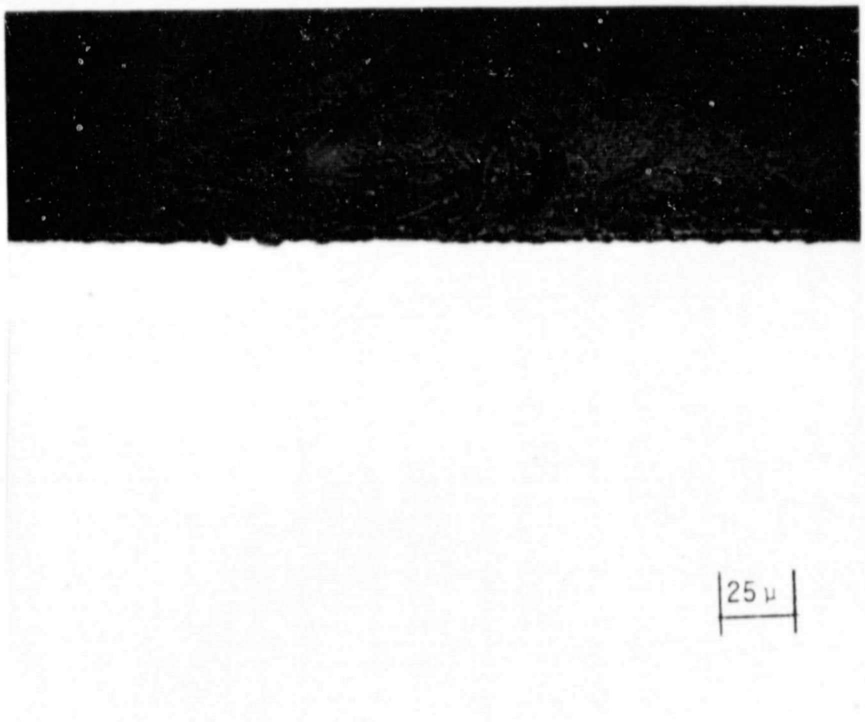
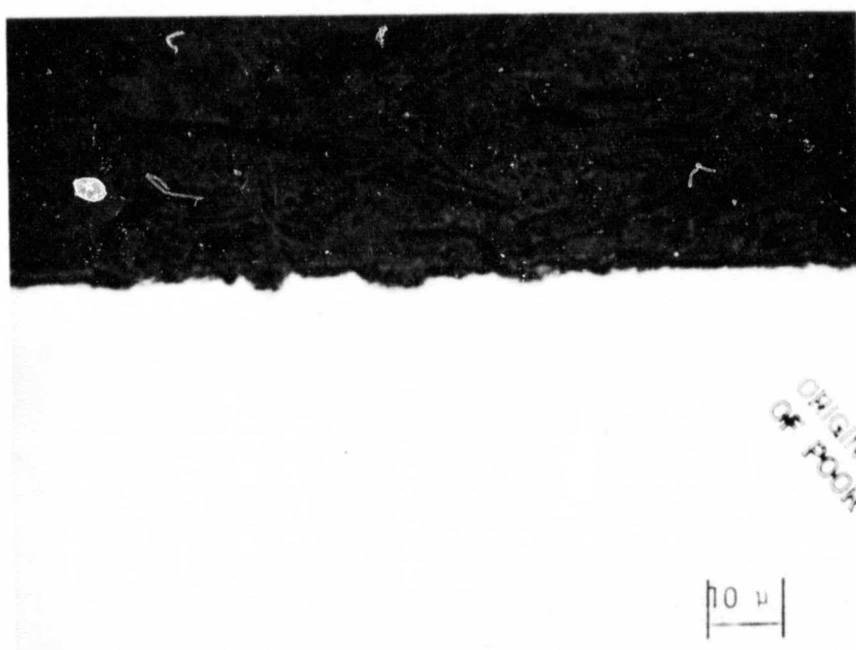


Figure G-3. Specimen Appearances After 39-Month F2 Immersion (Continued)

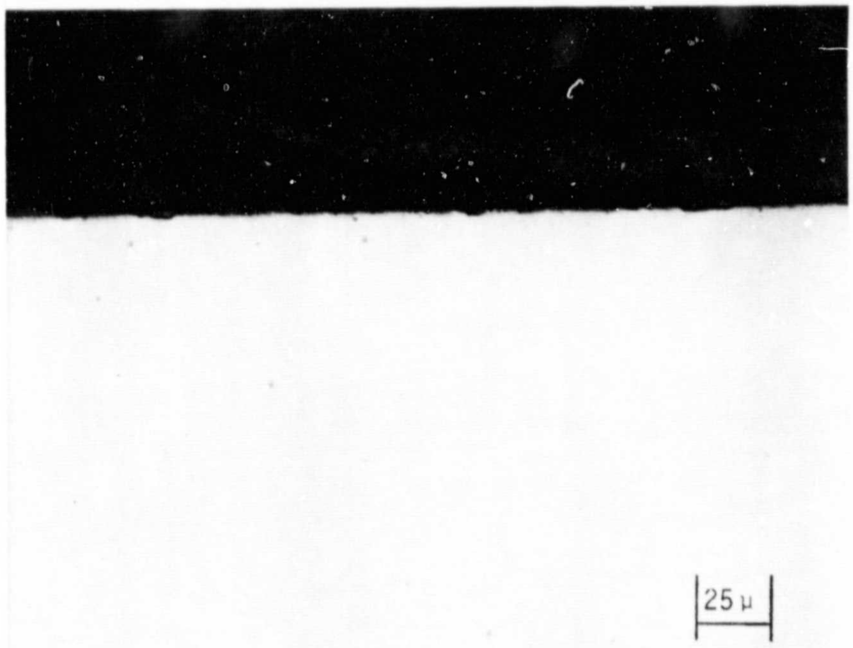


a) 7504 Vapor (∿ 400 X)

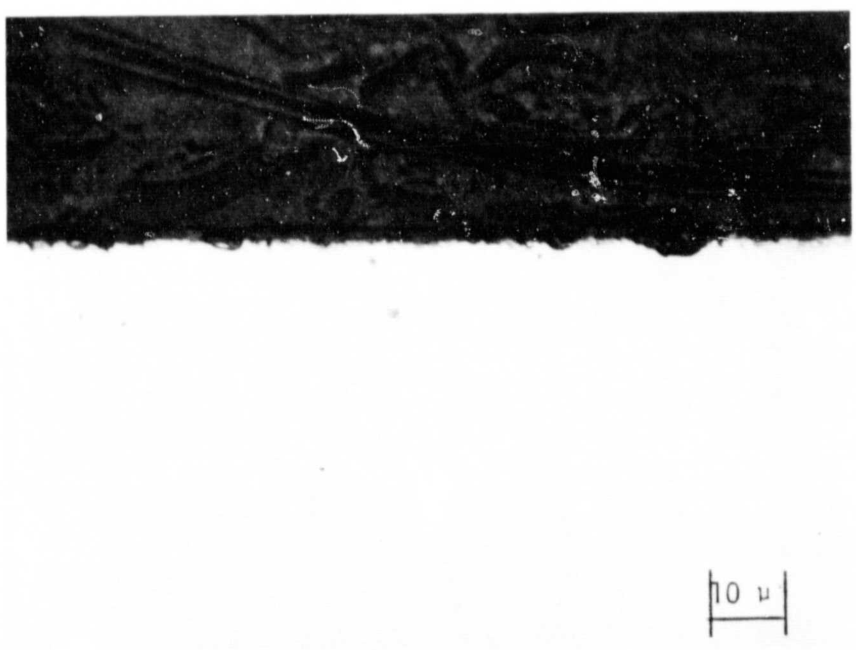


b) 7504 Vapor (∿1000 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs

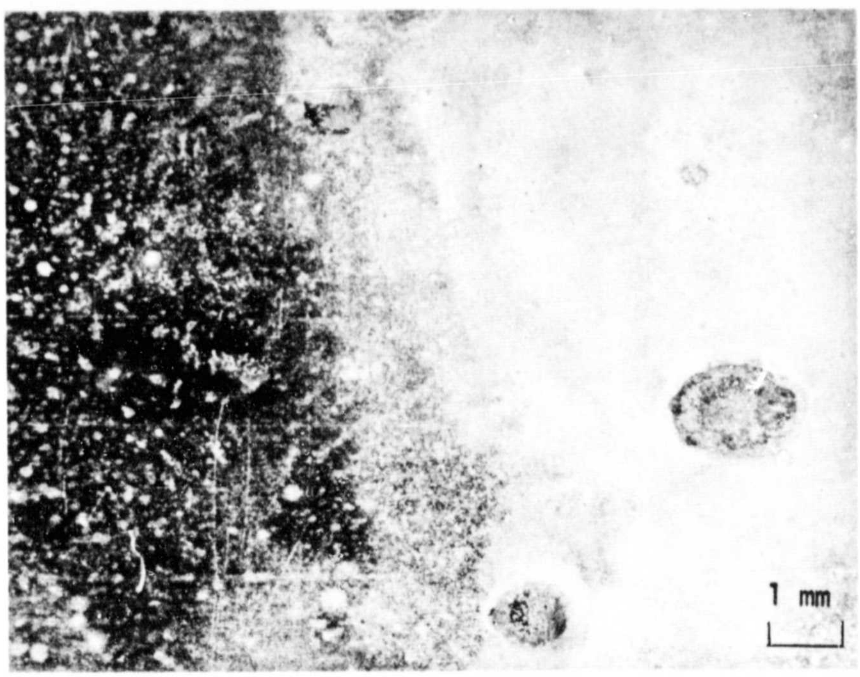


c) 7504 Liquid (∿ 400 X)



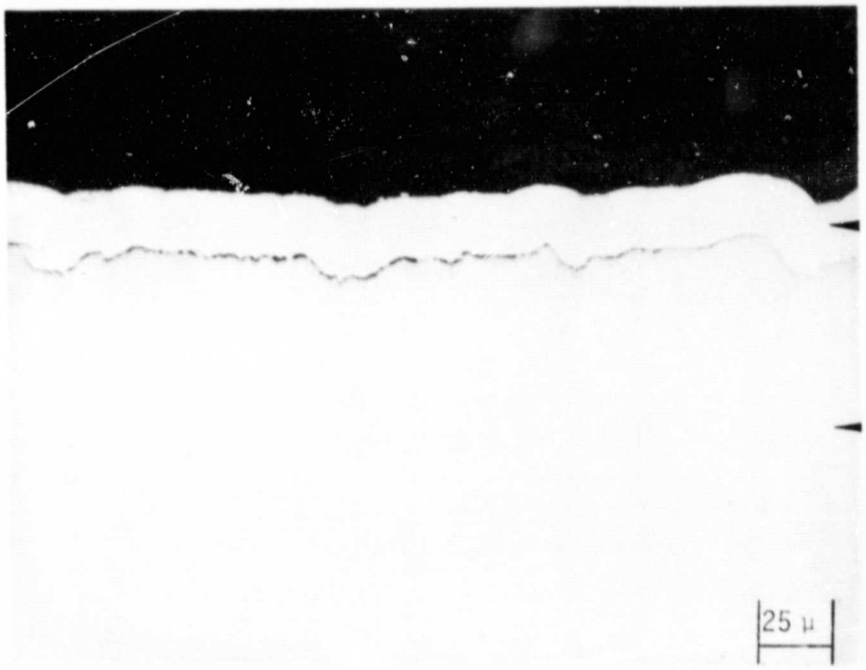
d) 7504 Liquid (∿ 1000 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)

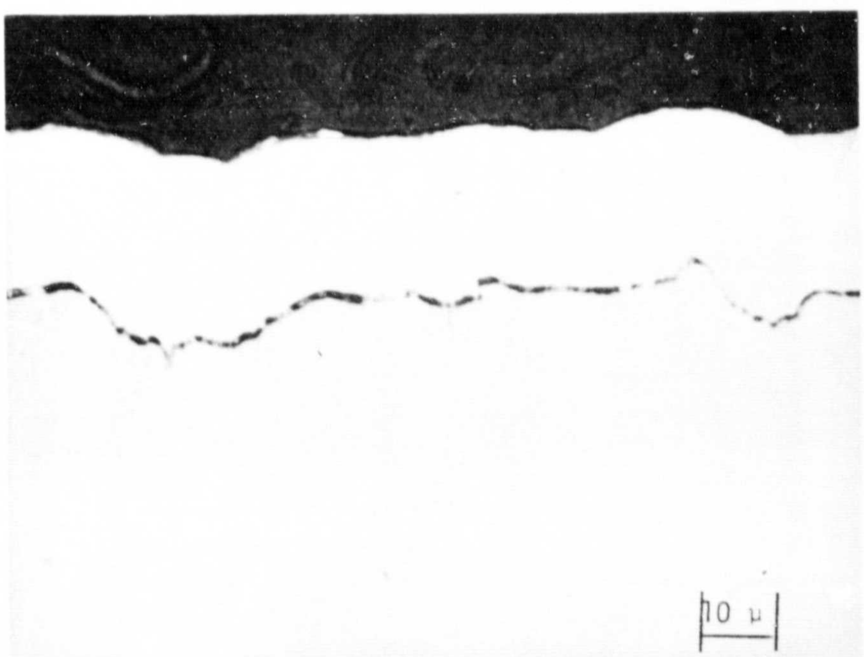


e) 7506 Vapor (~10 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)

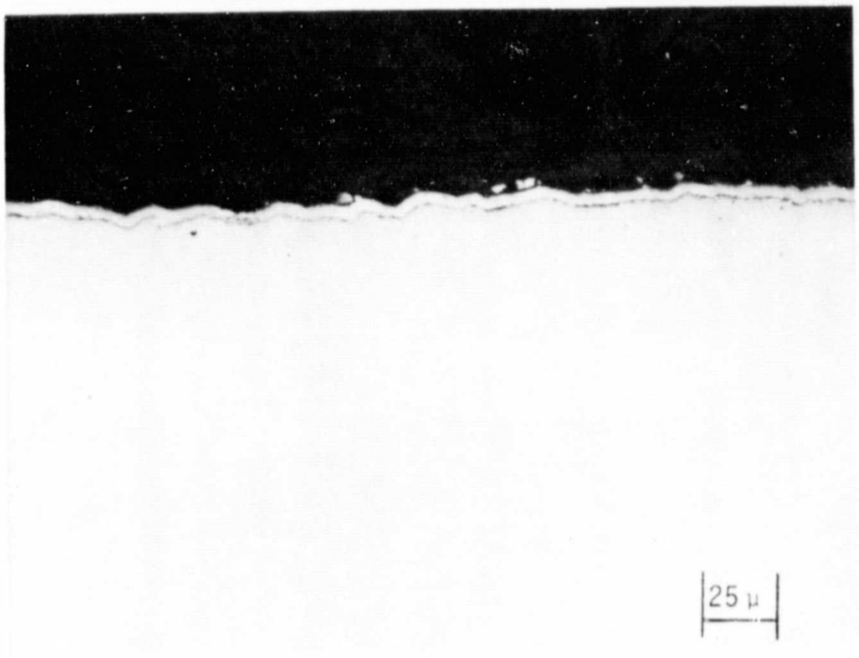


f) 7524 Vapor (∿ 400 X)

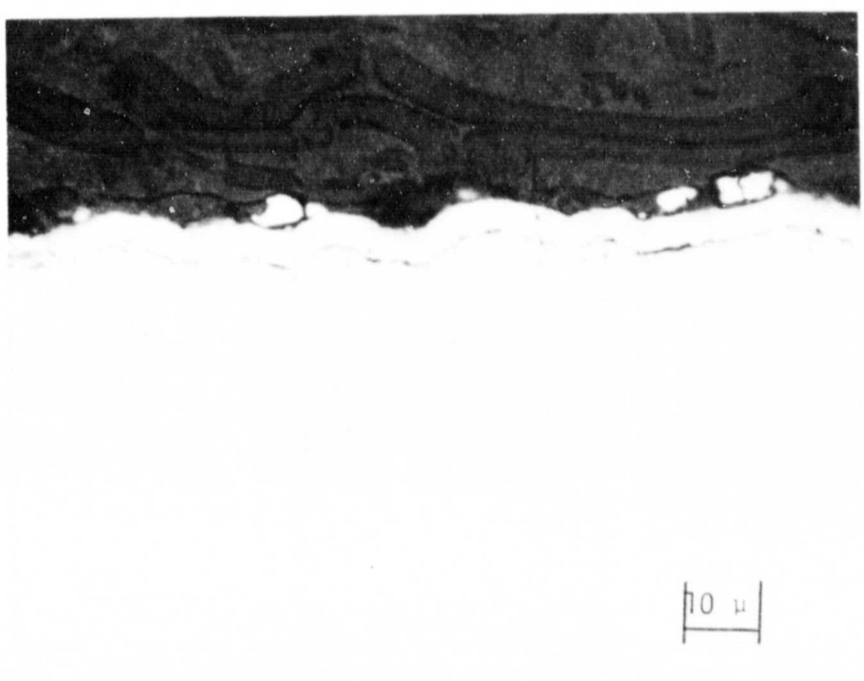


g) 7524 Vapor (∿ 1000 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)

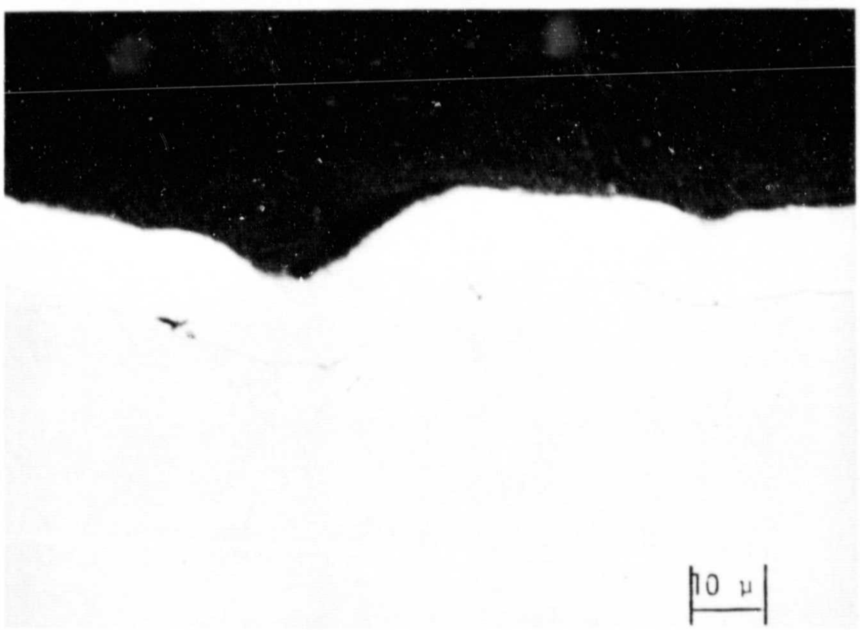


h) 7524 Liquid (~400 X)



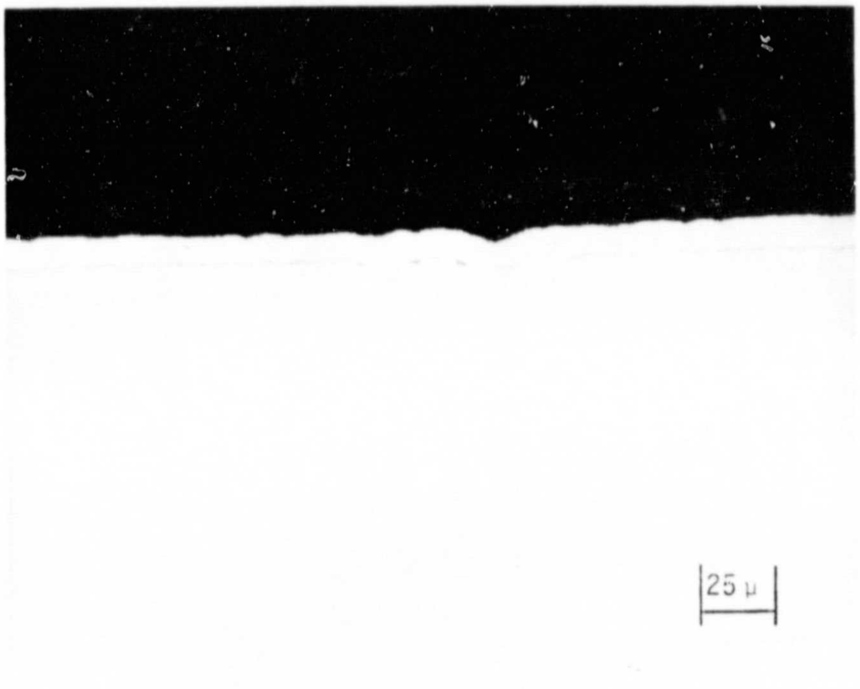
i) 7524 Liquid (~1000 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)



j) 7554 Vapor (~ 1000 X)

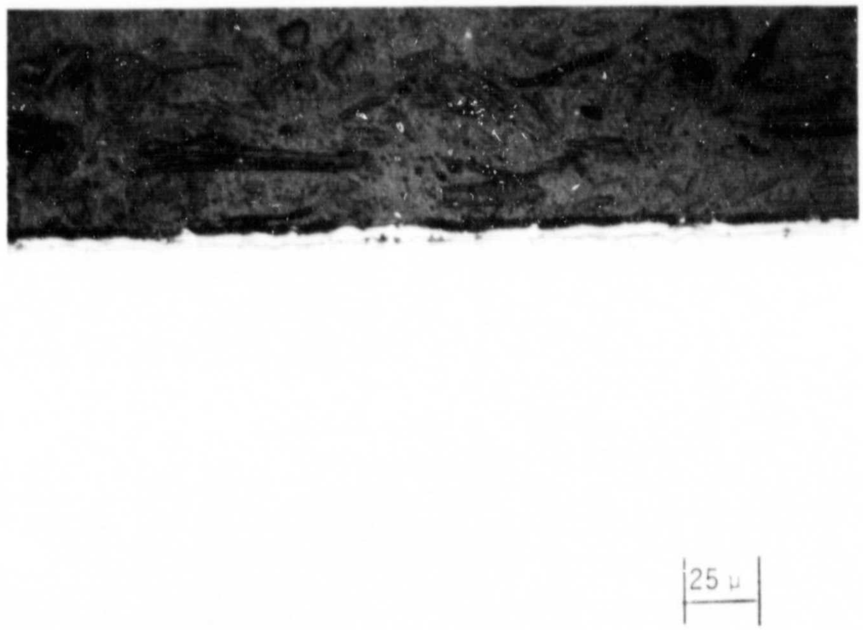
Figure G-4: 39-Month Immersion Post Exposure Photomicrographs (Continued)



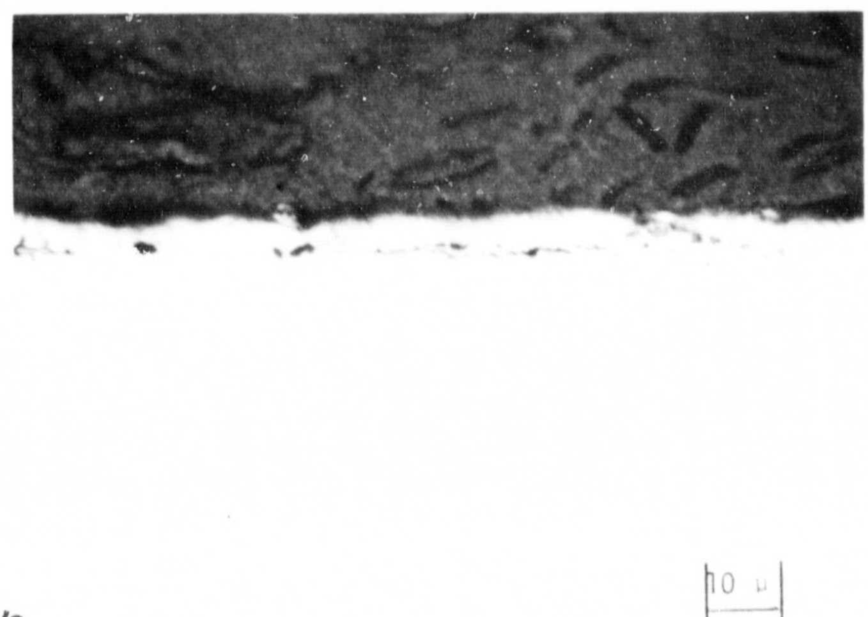
k) 7556 (~ 400 X)

l) 7556 Vapor (~ 1000 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)

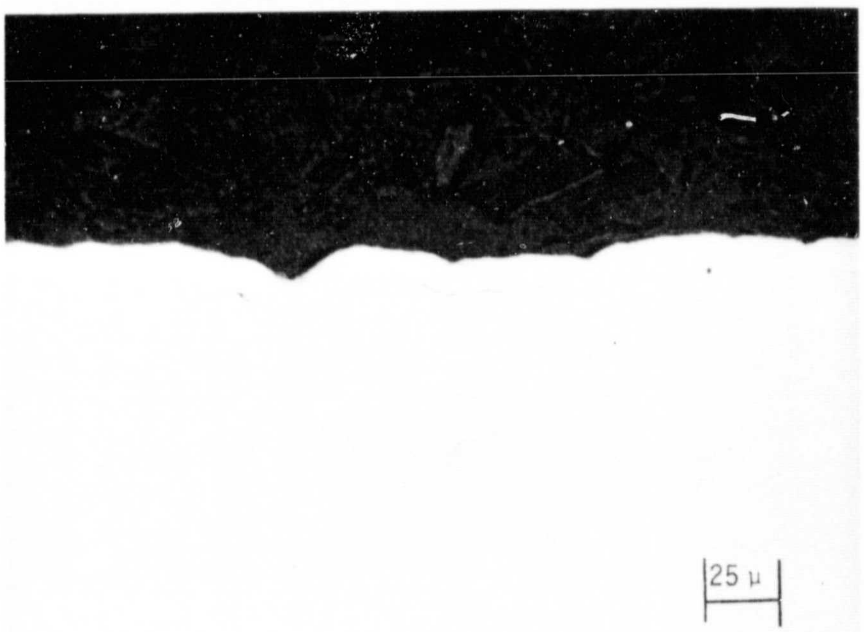


m) 7556 Liquid (~400 X)



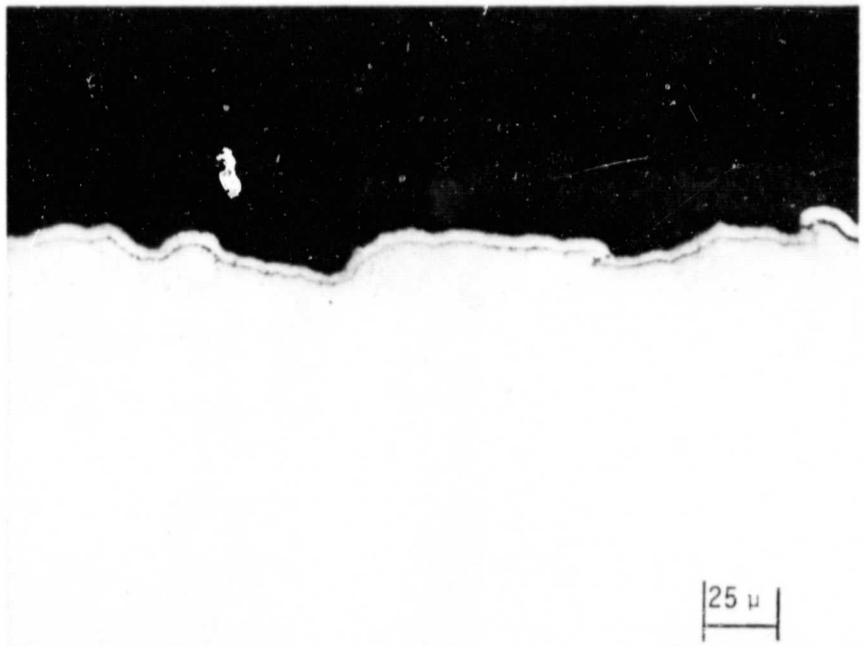
n) 7556 Liquid (~1000 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)

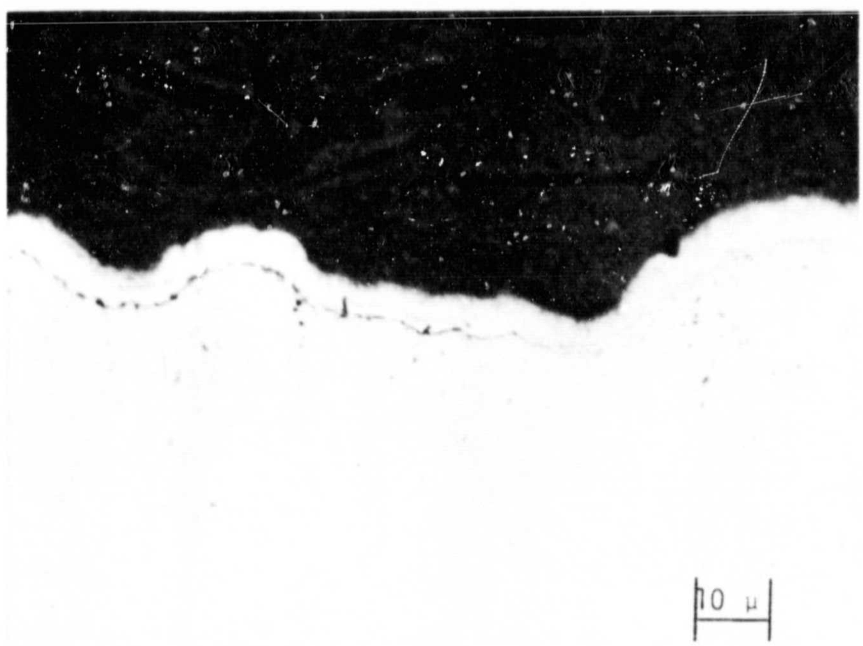


o) 7474 Vapor (~400 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)



p) 7574 Liquid (~400 X)



q) 7574 Liquid (~ 1000 X)

Figure G-4: 39-Month Immersion Post-Exposure Photomicrographs (Continued)

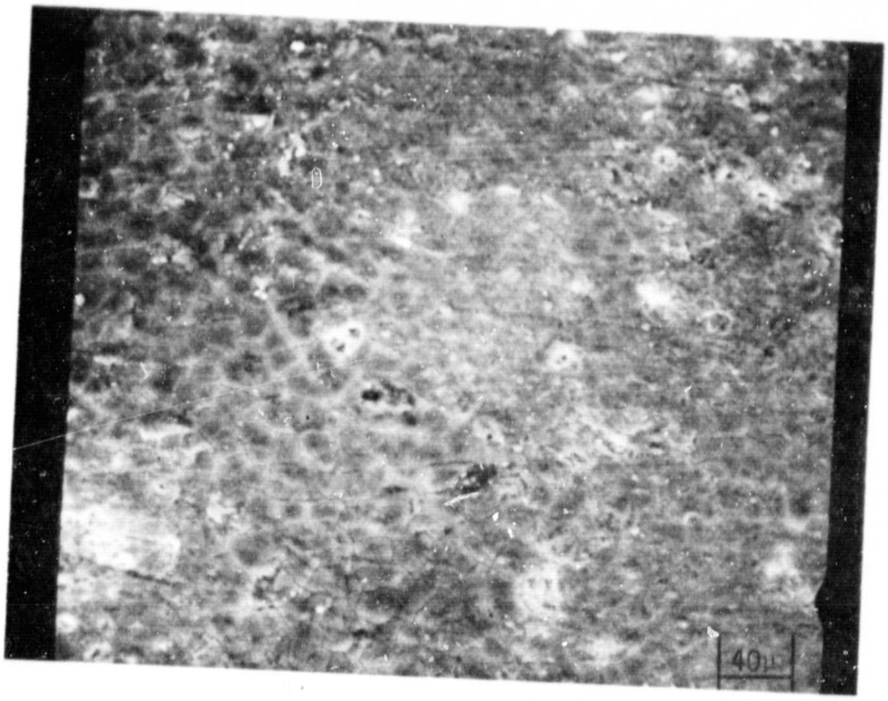


Figure G-5. Post-Exposure (39 Months) SEM Photograph, Specimen 7504 Vapor

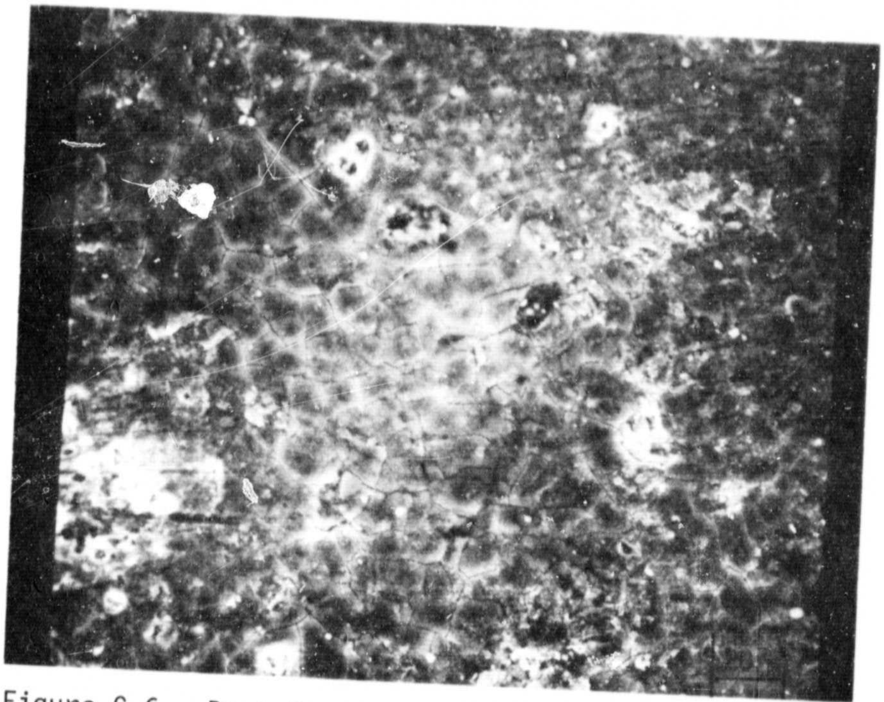


Figure G-6. Post Exposure (39 Months) SEM Photograph, Specimen 7504 Vapor

Figure G-7. Post Exposure (39 Months) SEM Photograph, Specimen 7504 Liquid

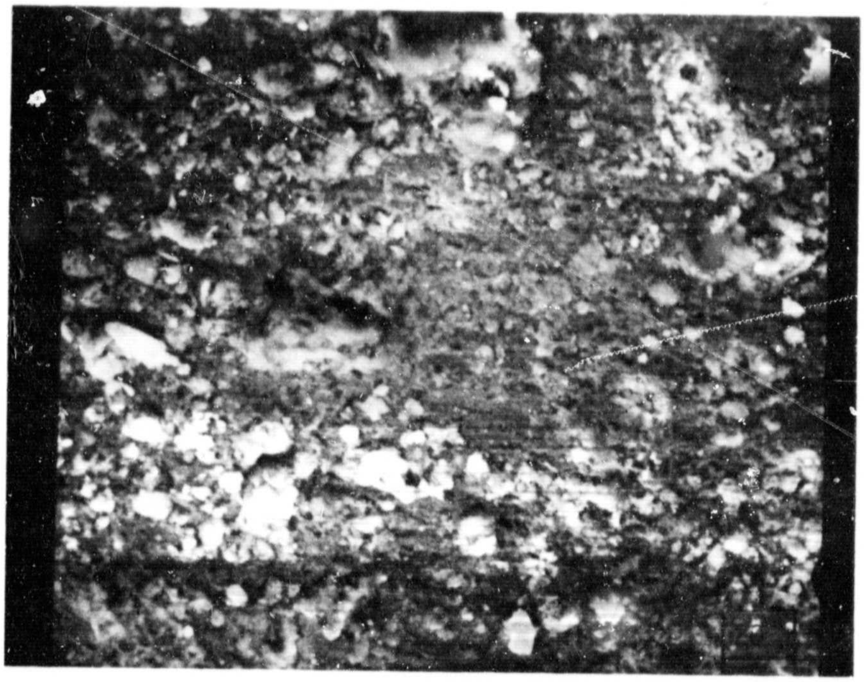


Figure G-8. Post Exposure (39 Months) SEM Photograph, Specimen 7504 Liquid

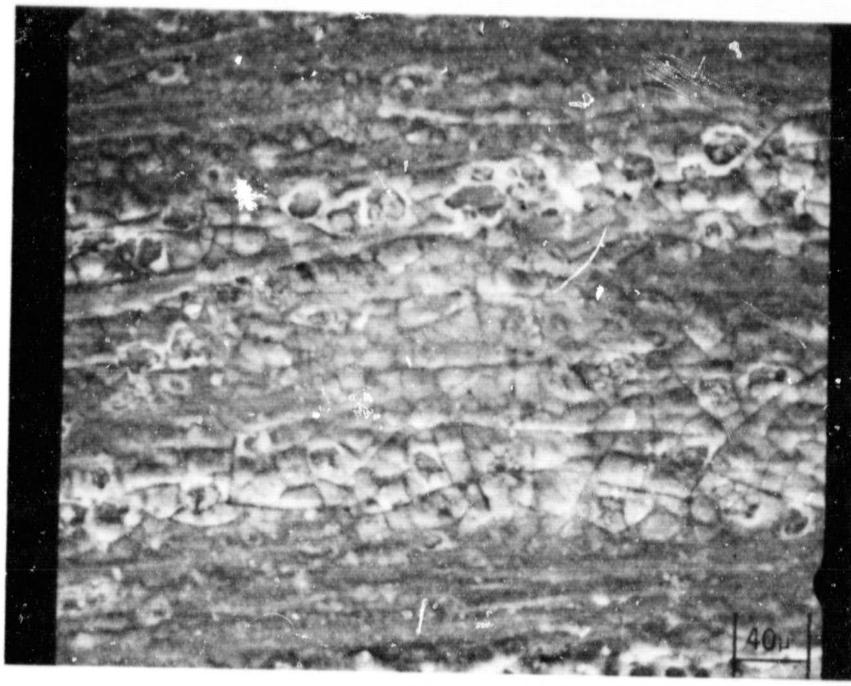


Figure G-9. Post Exposure (39 Months) SEM Photograph, Specimen 7523 Vapor

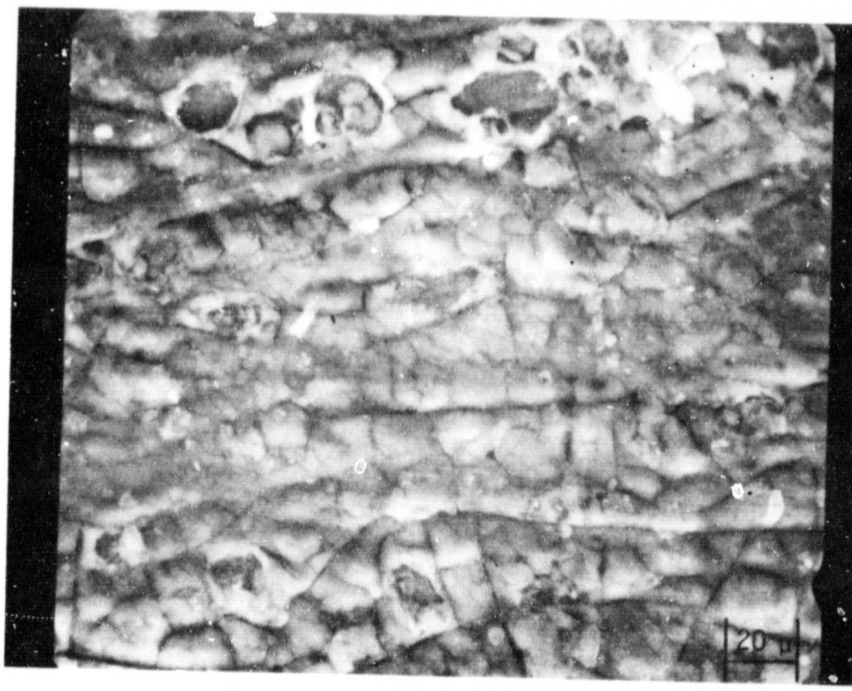


Figure G-10. Post Exposure (39 Months) SEM Photograph, Specimen 7523 Vapor

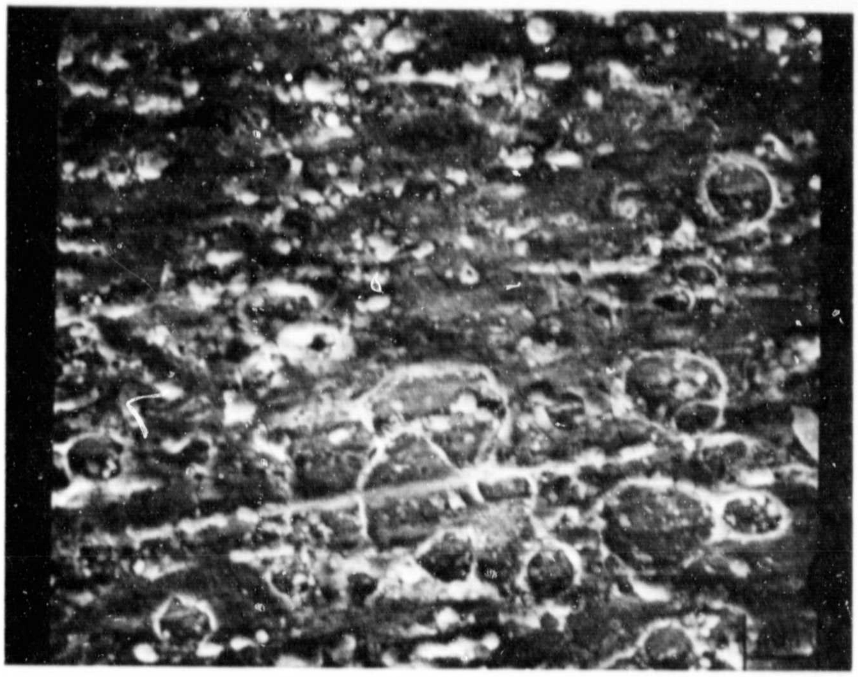


Figure G-11. Post Exposure (39 Months) SEM Photograph, Specimen 7523 Liquid

Figure G-12. Post Exposure (39 Months) SEM Photograph, Specimen 7523 Liquid

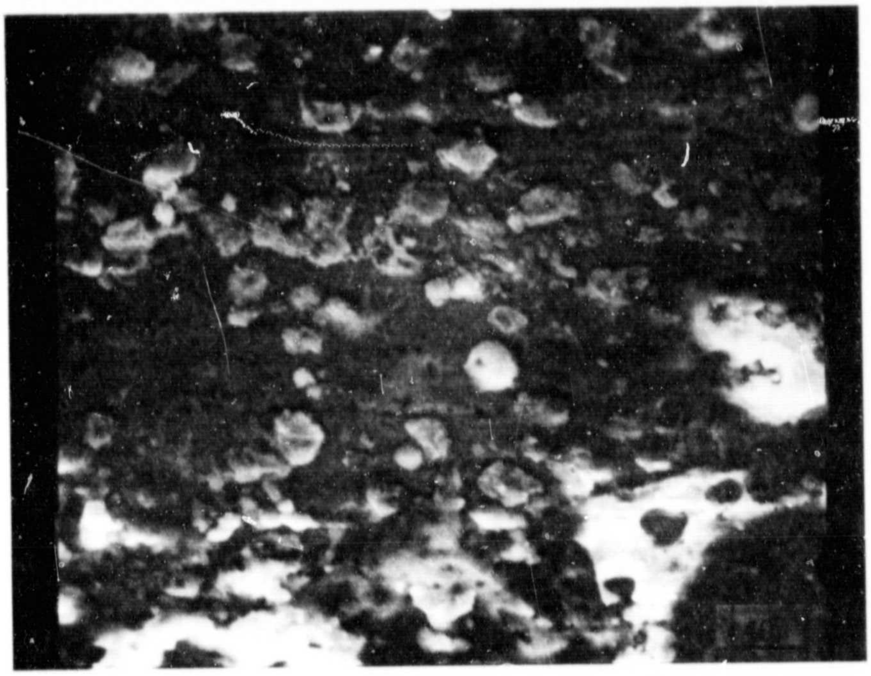


Figure G-13. Post-Exposure (39-Months) SEM Photograph, Specimen 7554 Liquid

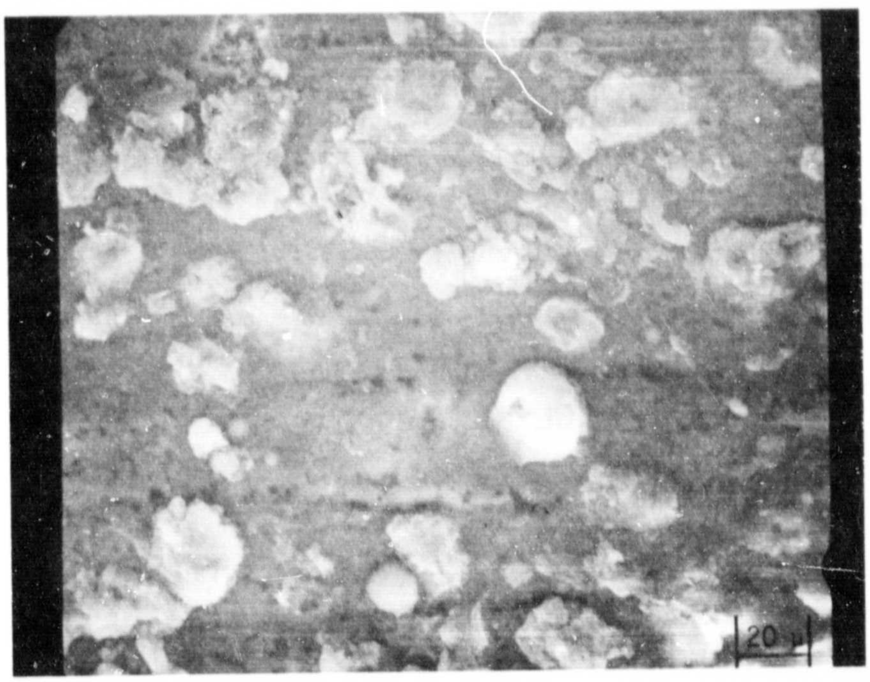


Figure G-14. Post-Exposure (39-Months) SEM Photograph, Specimen 7554 Liquid

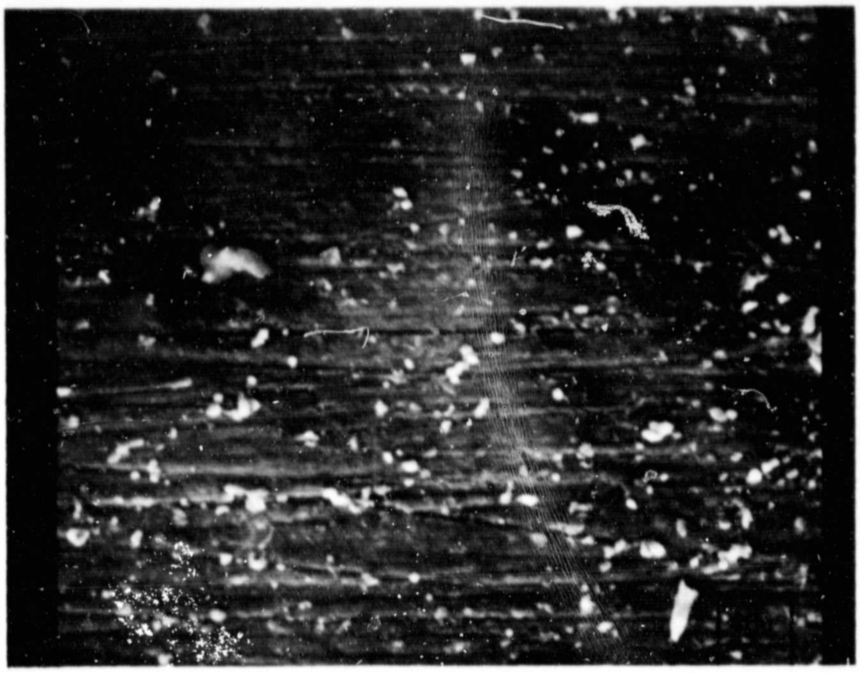


Figure G-15. Post-Exposure (39-Months) SEM Photograph, Specimen 7554 Vapor

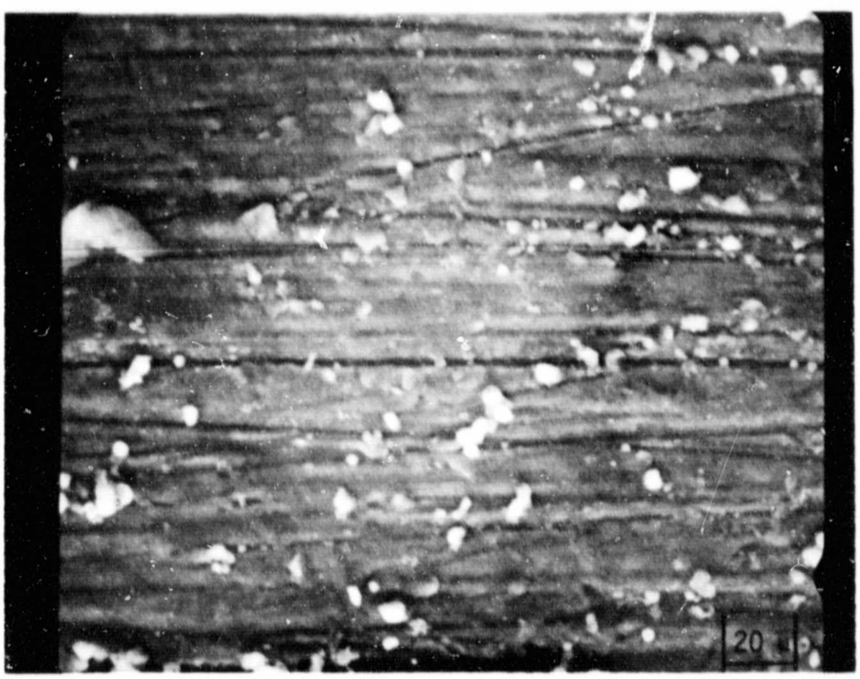


Figure G-16. Post-Exposure (39-Months) SEM Photograph, Specimen 7554 Vapor

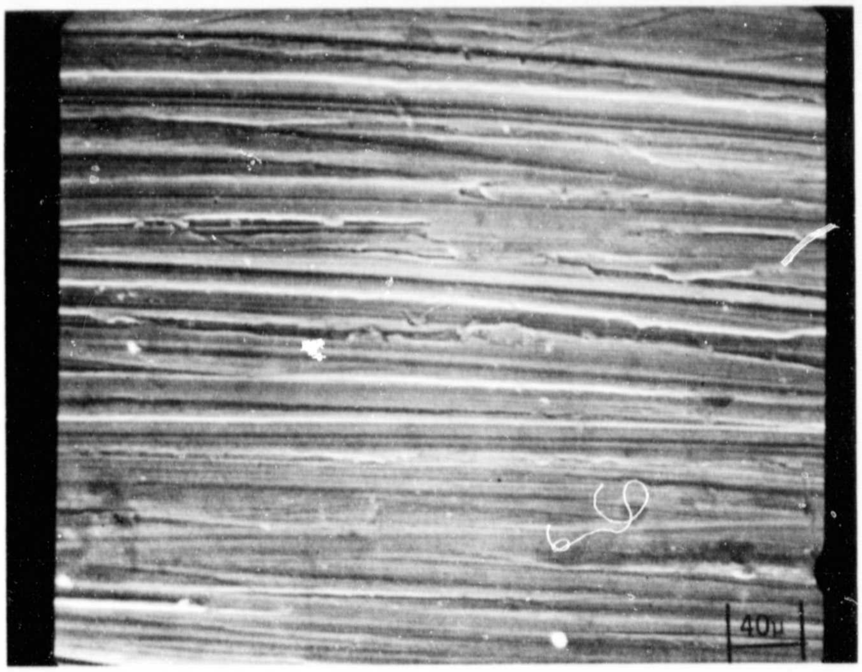


Figure G-17. Post Exposure (39 Months) SEM Photograph, Specimen 7573 Vapor

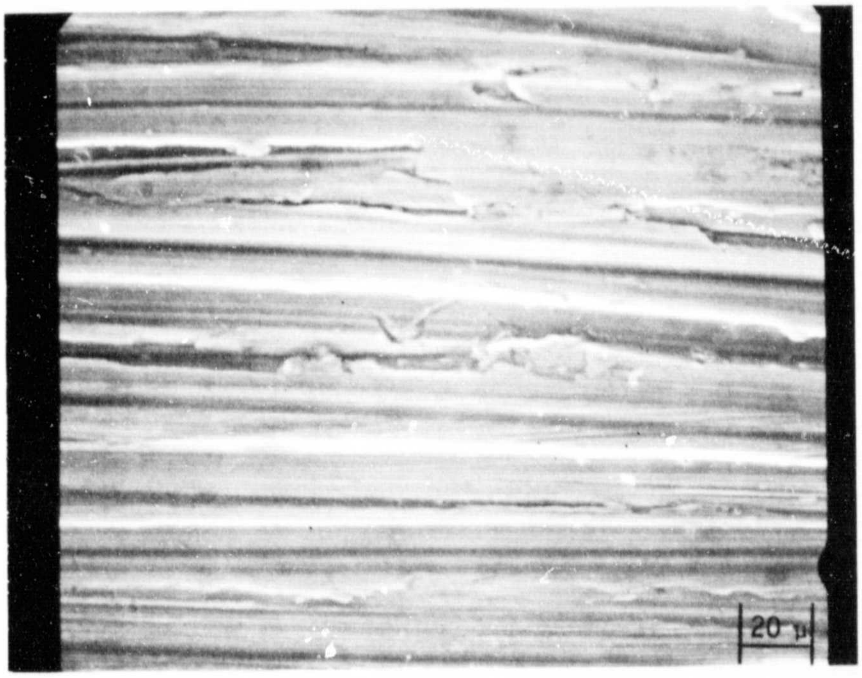


Figure G-18. Post Exposure (39 Months) SEM Photograph, Specimen 7573 Vapor

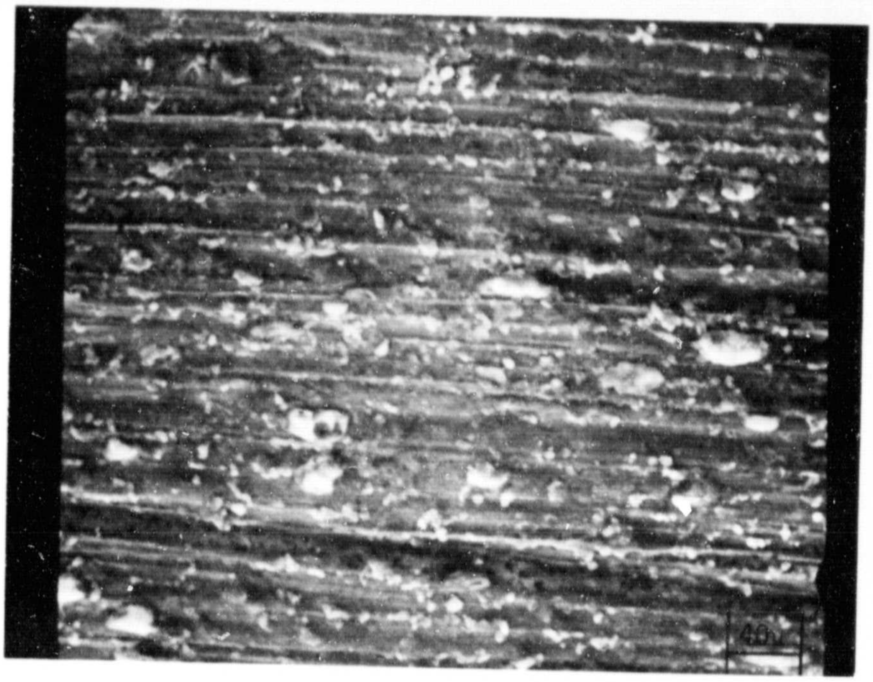


Figure G-19. Post Exposure (39 Months) SEM Photograph, Specimen 7573 Liquid

Figure G-20. Post Exposure (39 Months) SEM Photograph, Specimen 7573 Liquid

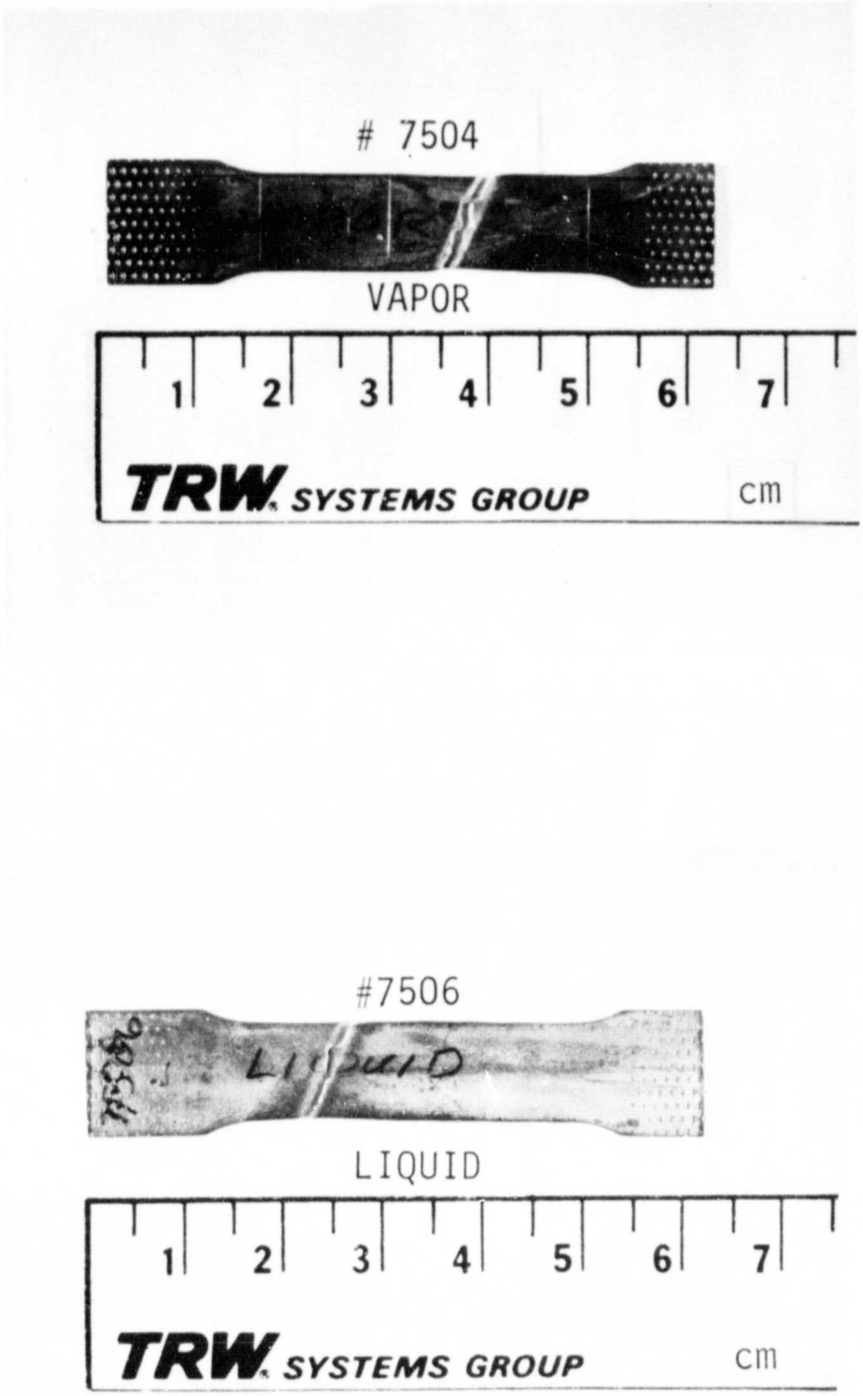
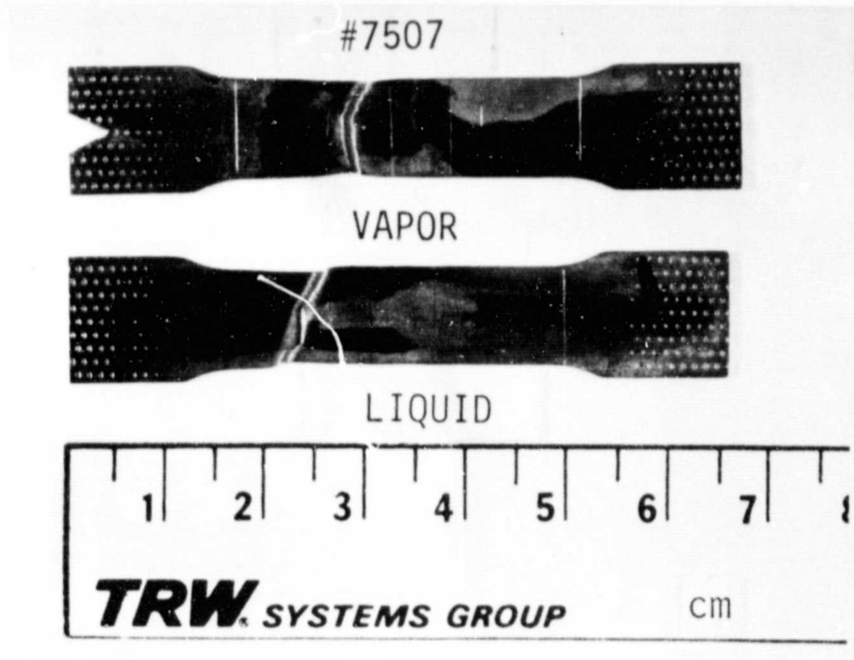


Figure G-21: Post Test Tensile Coupons 298 K



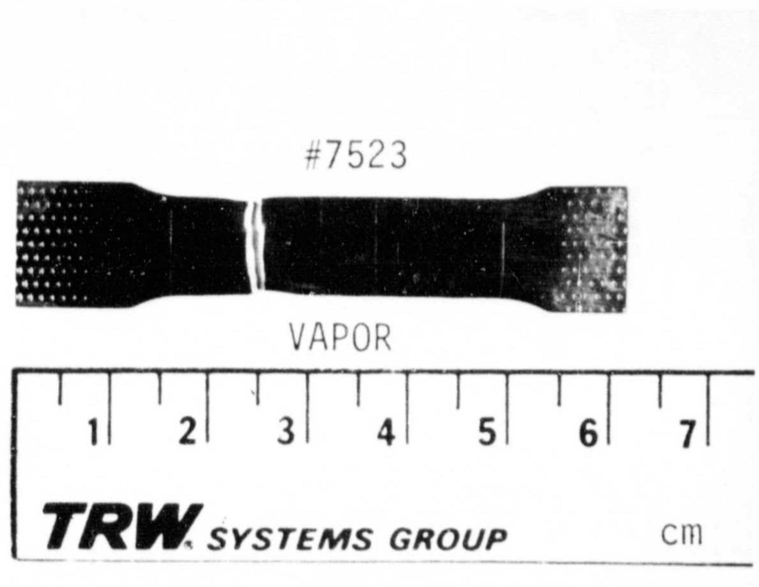
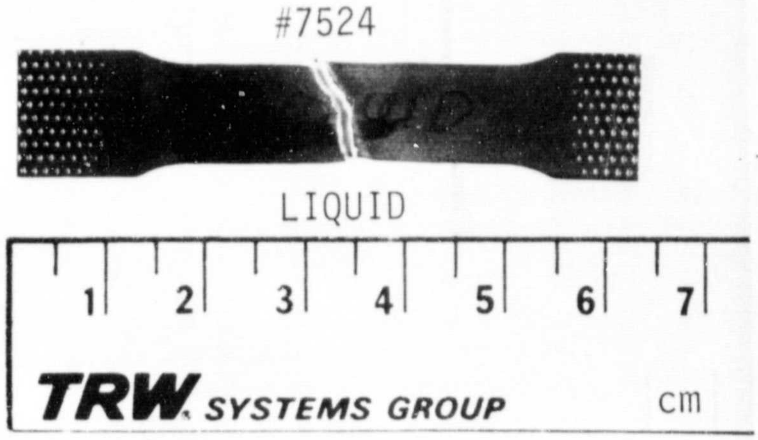


Figure G-21: Post-Test Tensile Coupons 298 K (Continued)



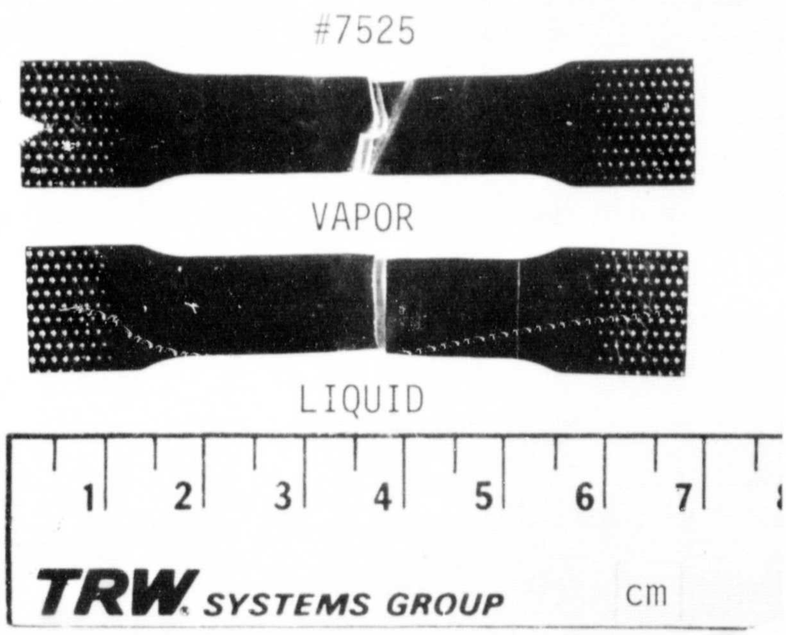
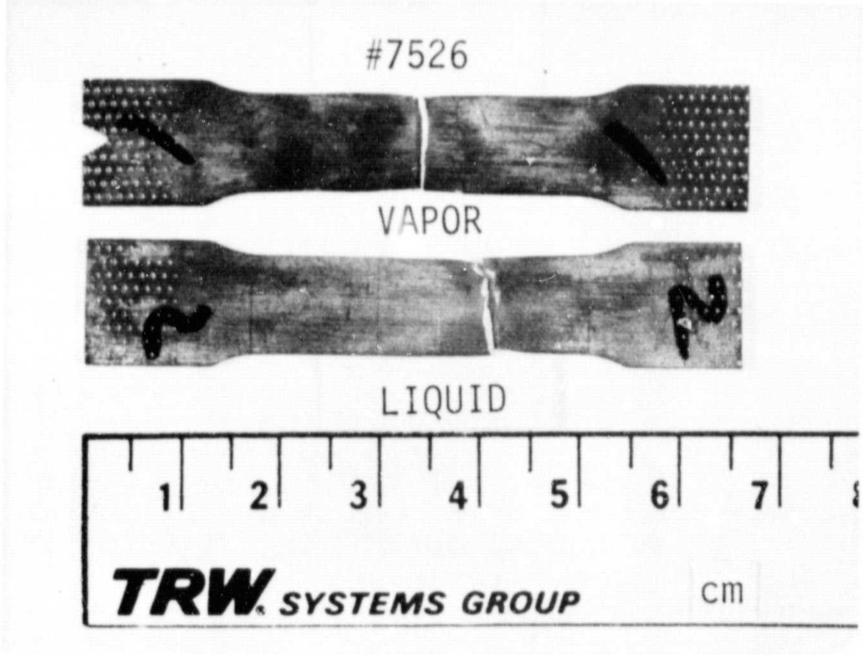


Figure G-21: Post-Test Tensile Coupons 298 K (Continued)



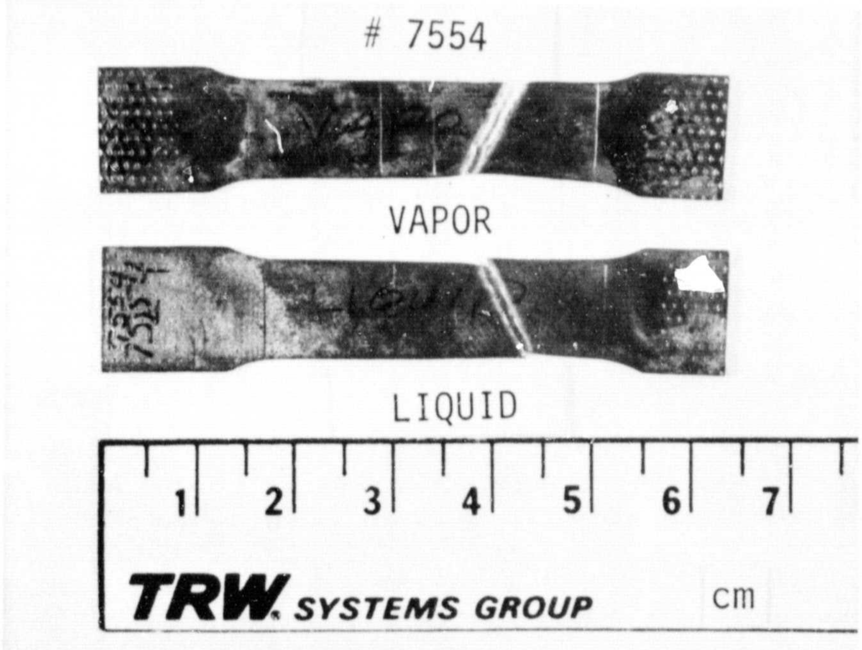
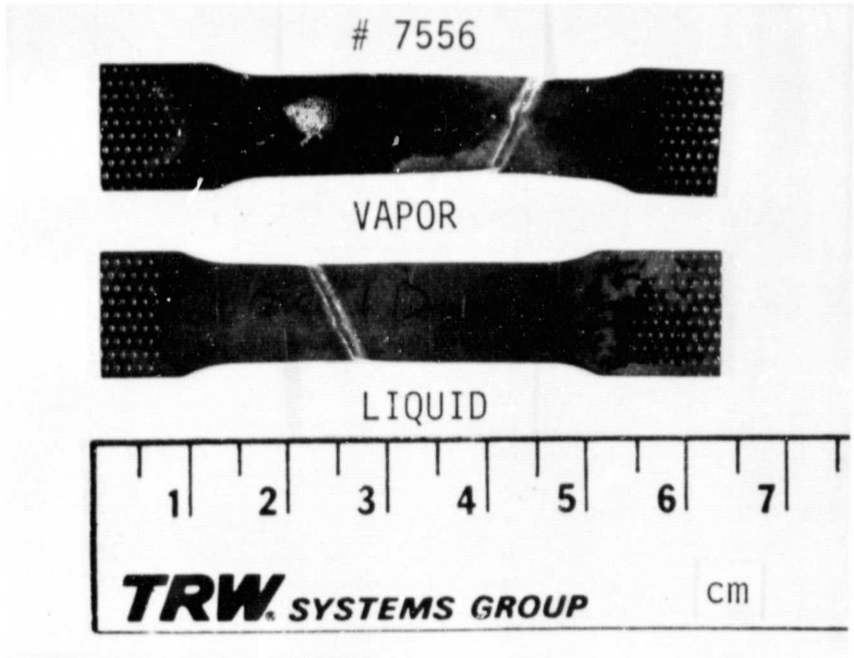


Figure G-21: Post-Test Tensile Coupons 298 K (Continued)



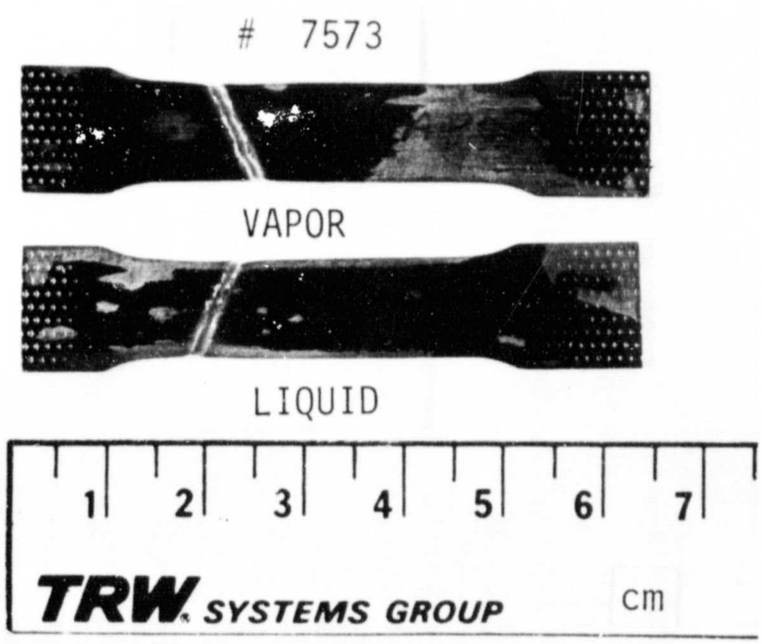


Figure G-21: Post-Test Tensile Coupons 298 K (Continued)

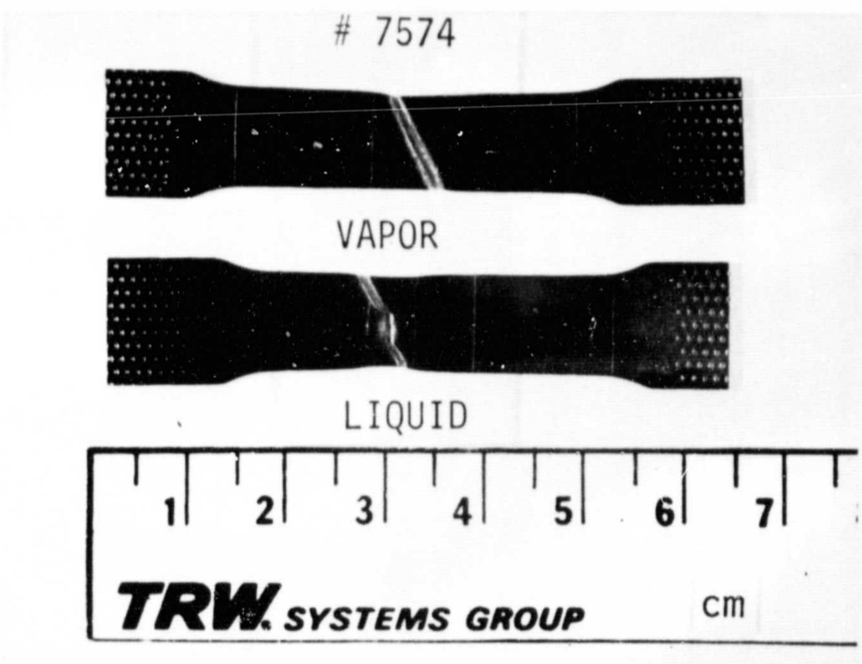
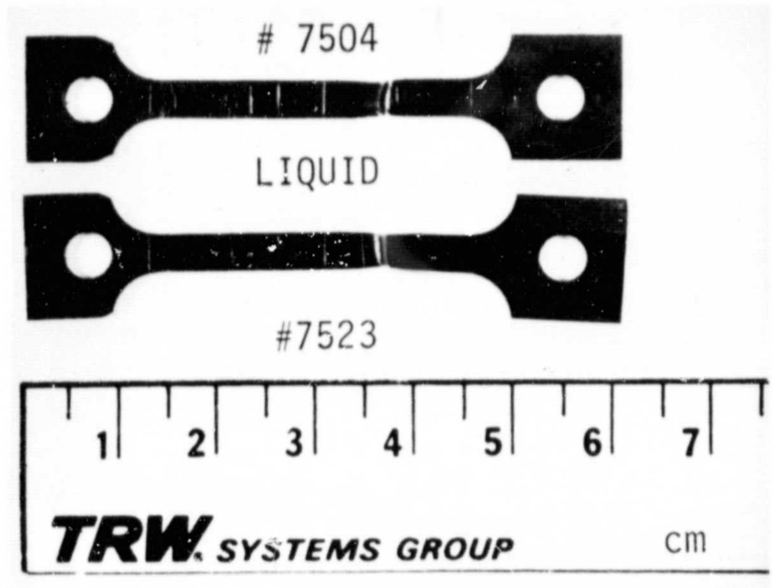


Figure G-21: Post-Test Tensile Coupons 298 K (Continued)



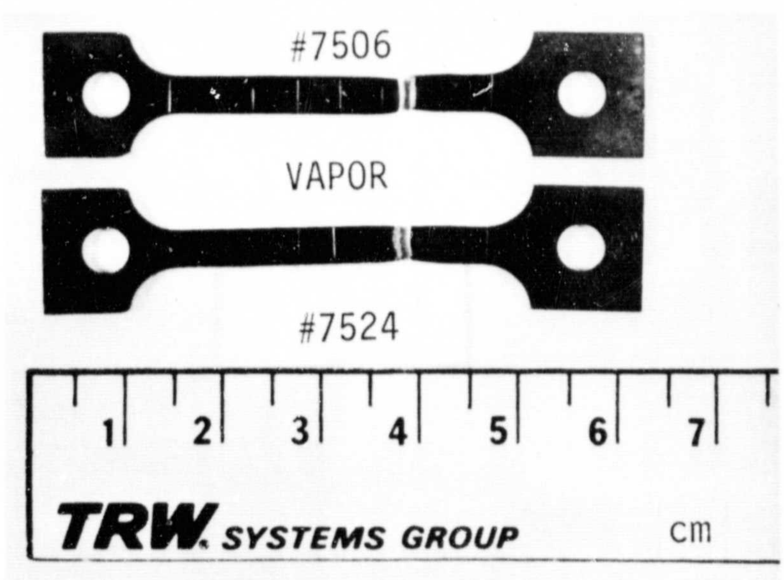
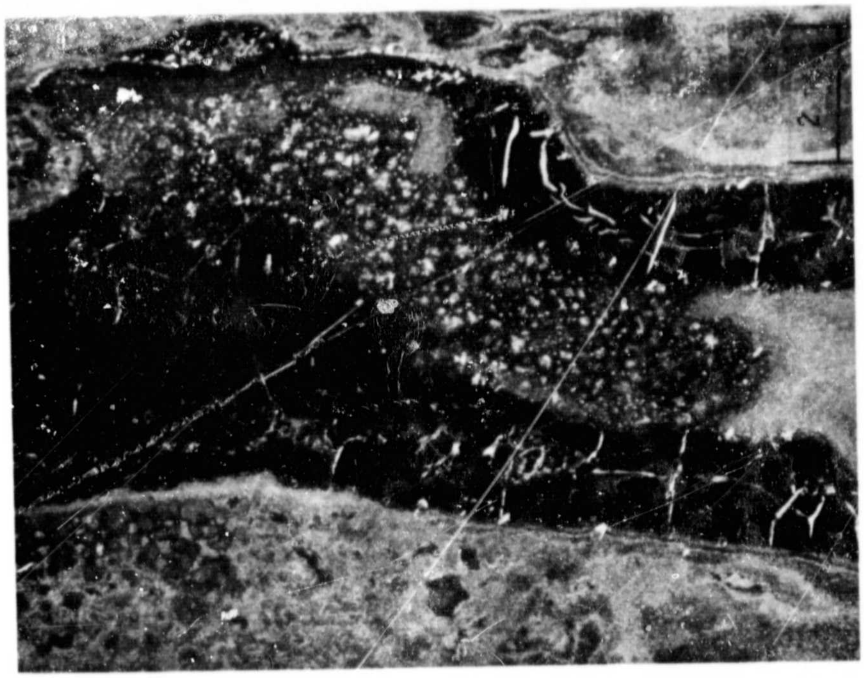
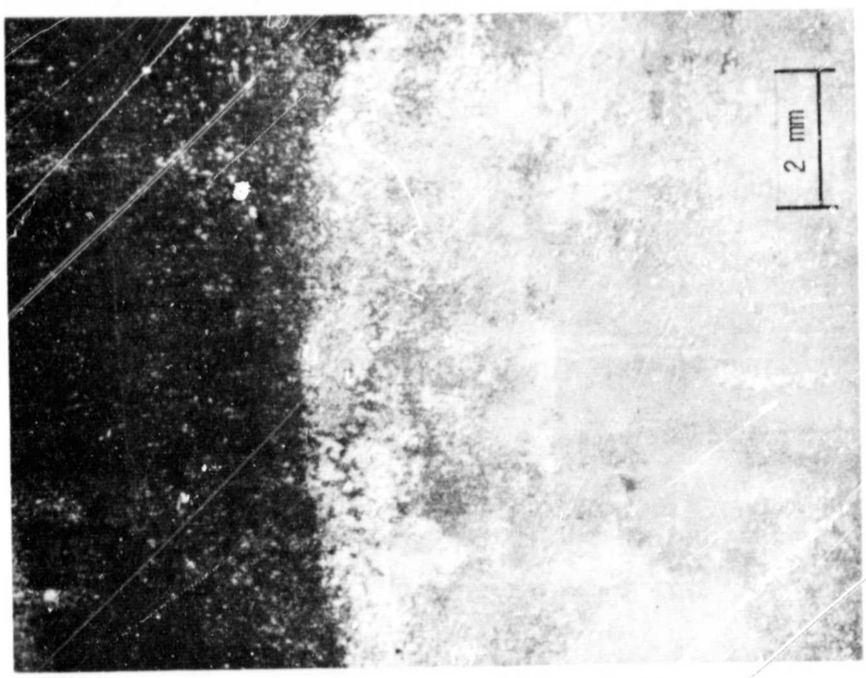


Figure G-22. Post-Test Tensile Coupons 77 K

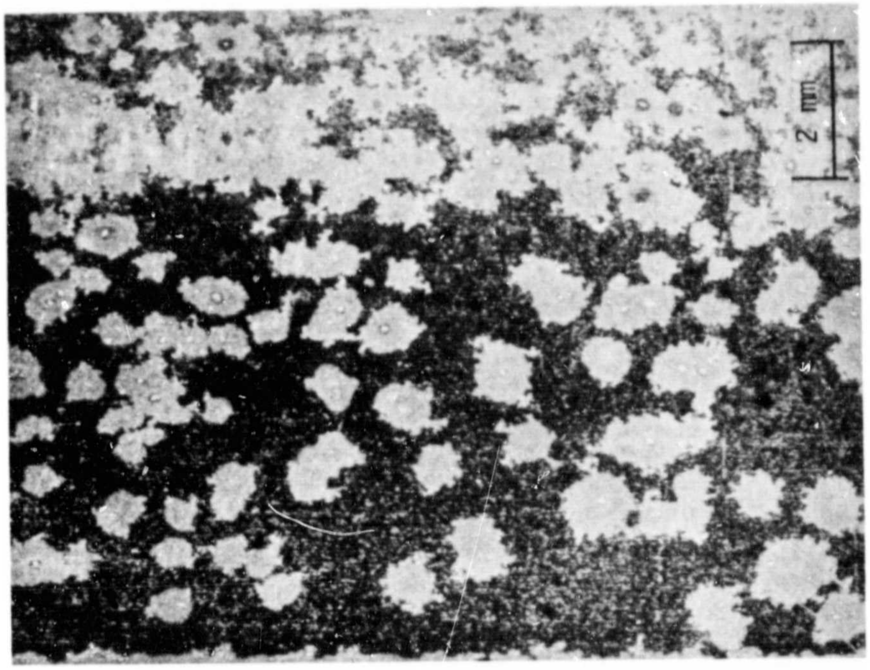


a) 7504, Vapor, Side 1

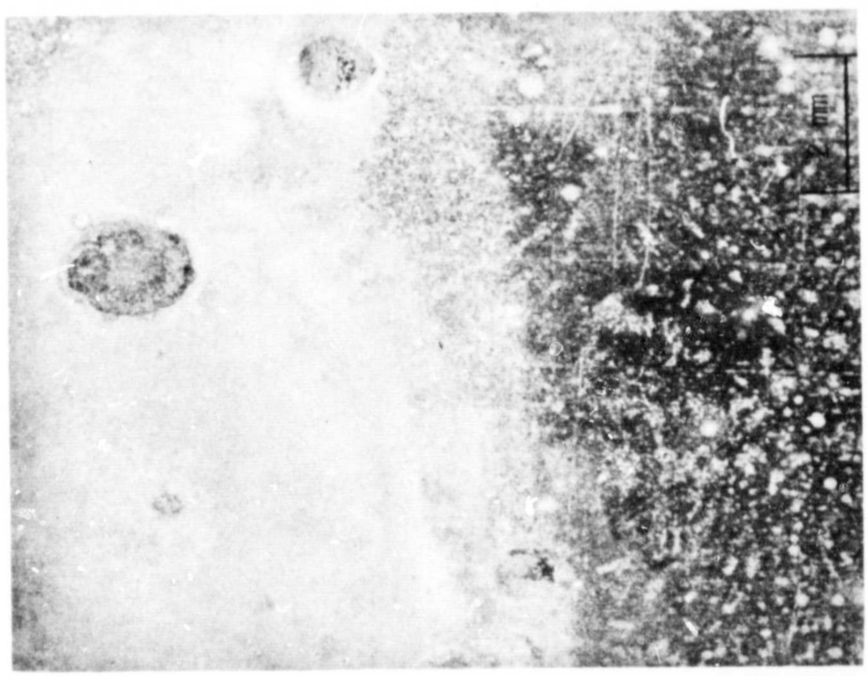


b) 7504, Liquid, Side 1

Figure G-23: Low Magnification Photographs of Surface Features, 39-Month Immersion (~10X)

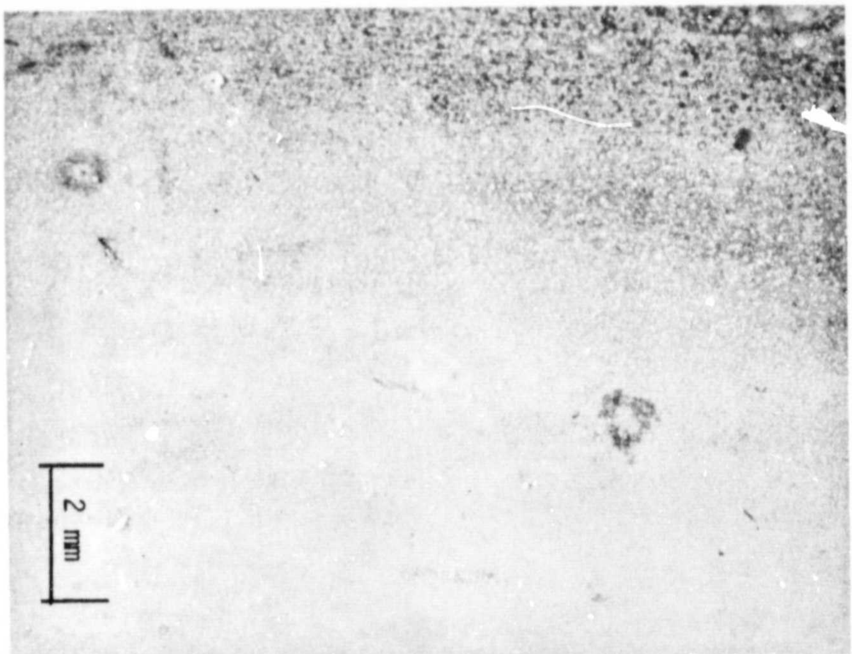


c) 7504, Liquid, Side 2

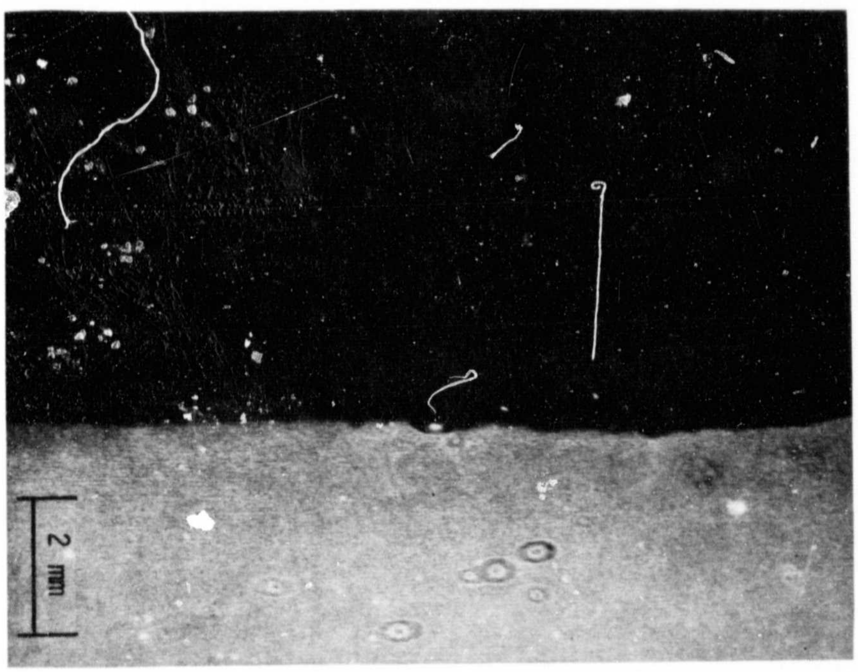


d) 7506, Vapor

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (∿10X)

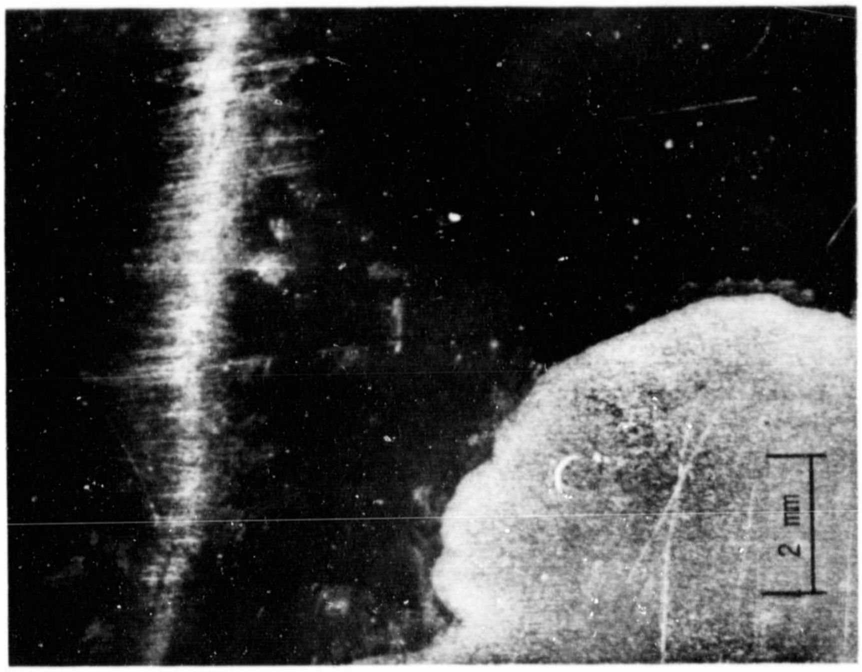


e) 7506, Liquid, Region 1



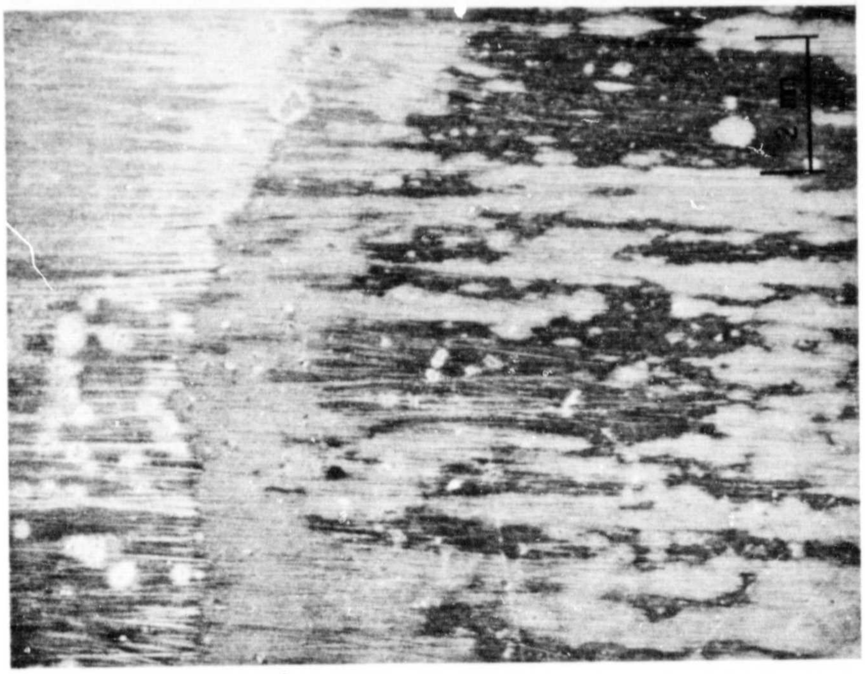
f) 7506, Liquid, Region 2

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion ($\sim$10X)

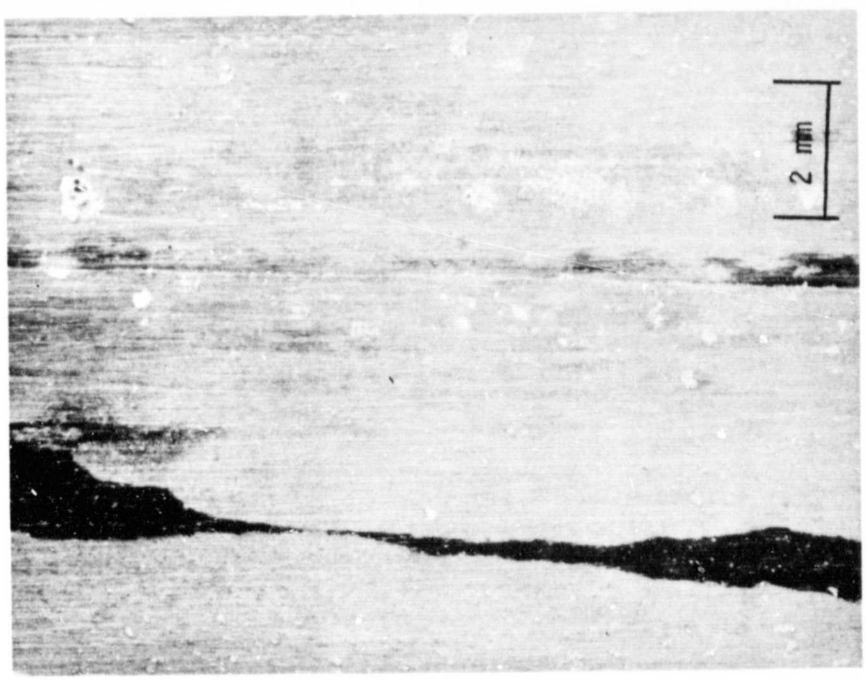


g) 7507, Vapor

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (~10X)

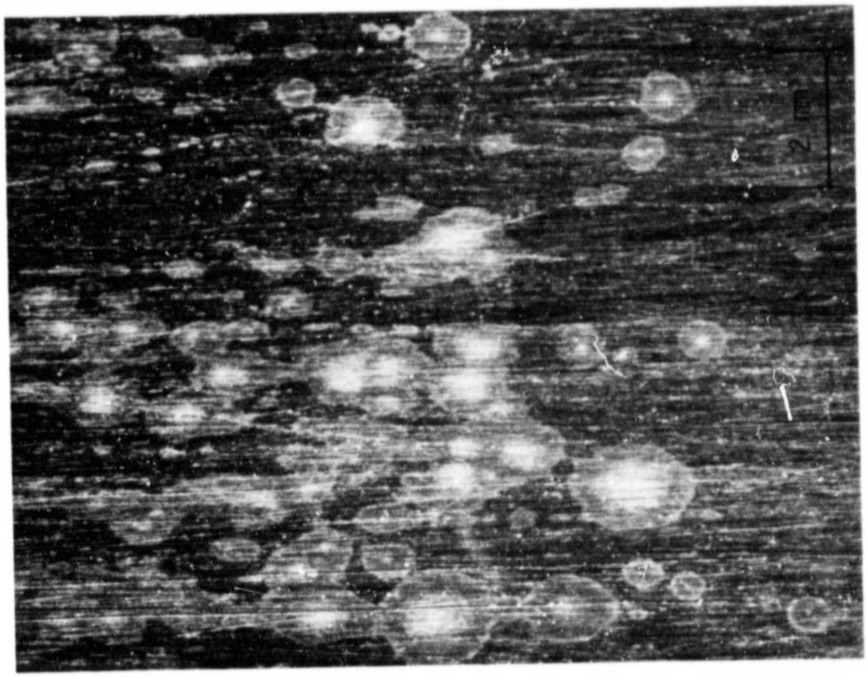


h) 7523, Vapor

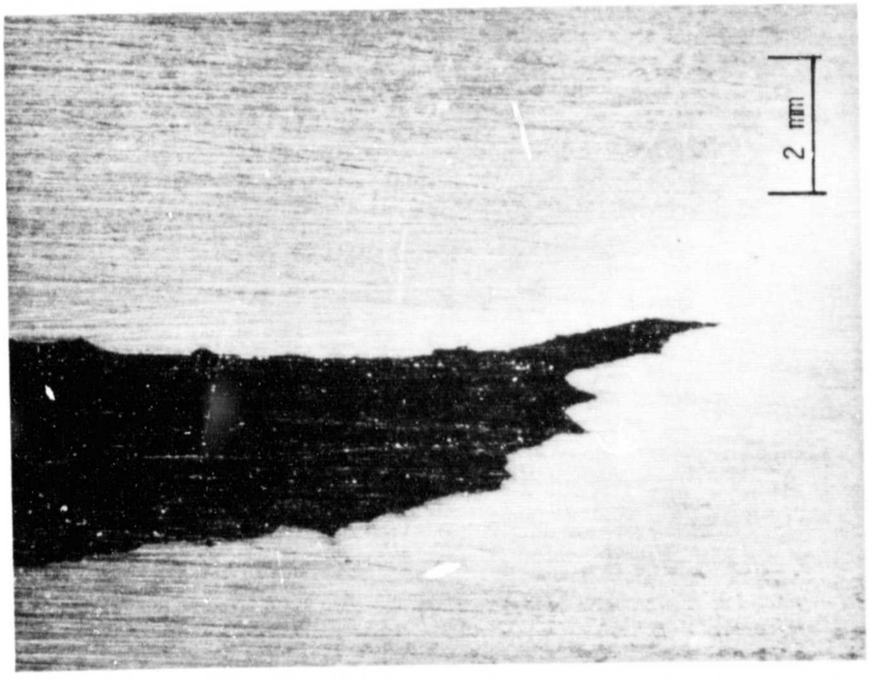


i) 7523, Liquid

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (~10X)

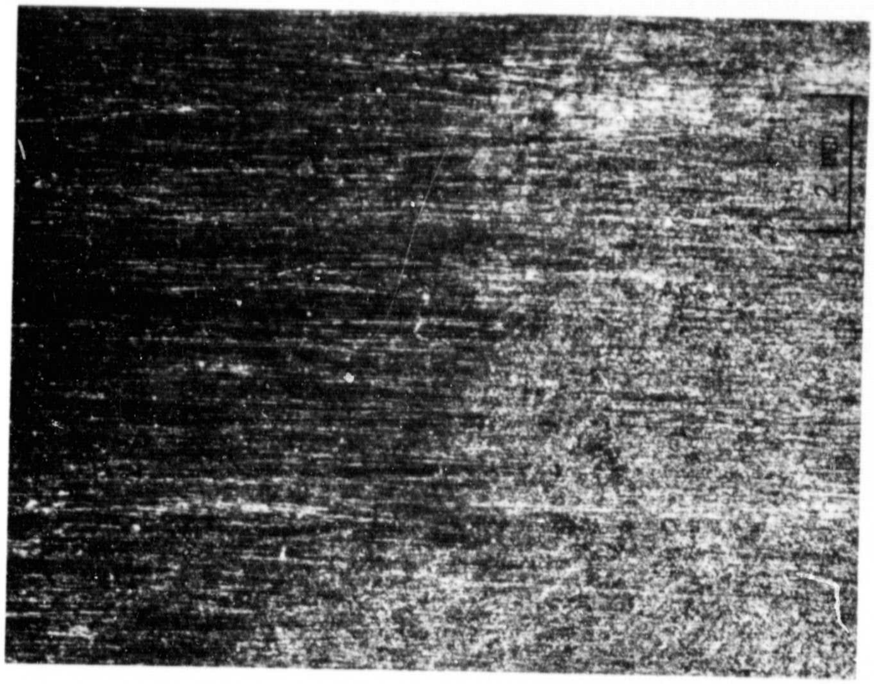


j) 7524, Vapor

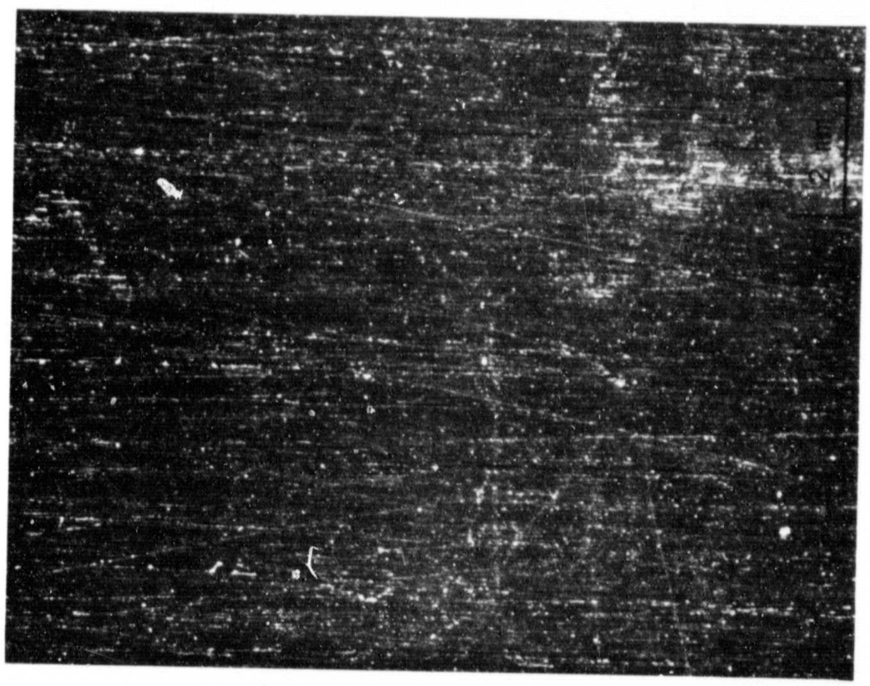


k) 7524, Liquid

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion ($\sim$10X)



l) 7525, Vapor



m) 7525, Liquid

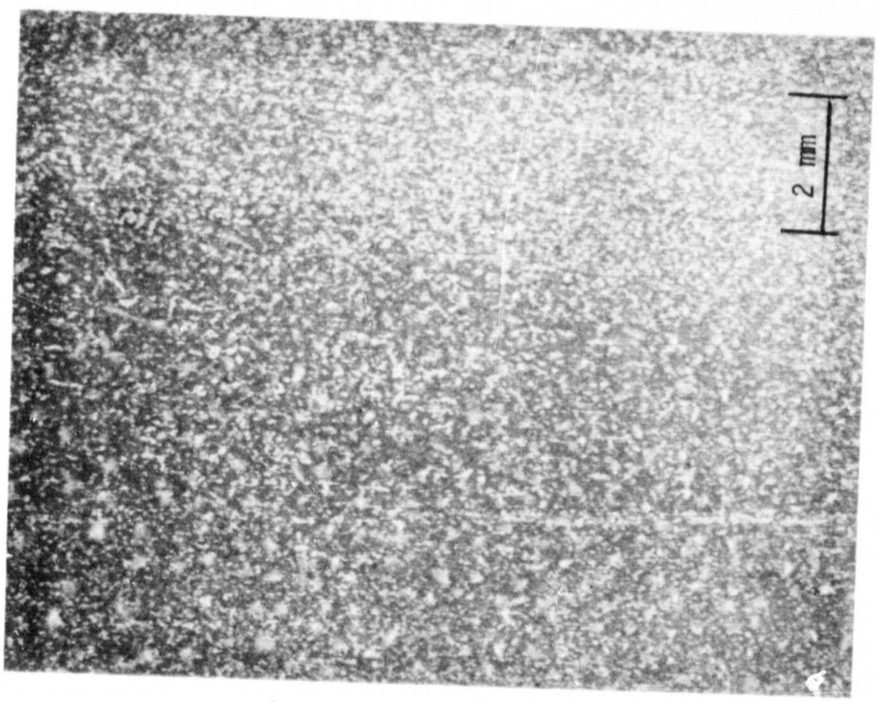
Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (∿10X)

n) 7526, Vapor

o) 7526, Liquid

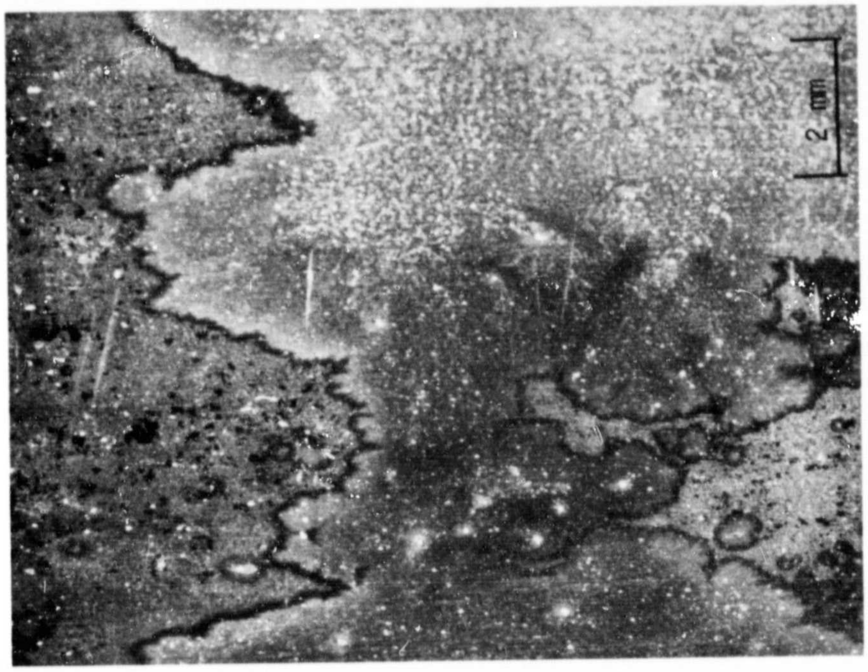
Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (∼10X)

p) 7554, Vapor

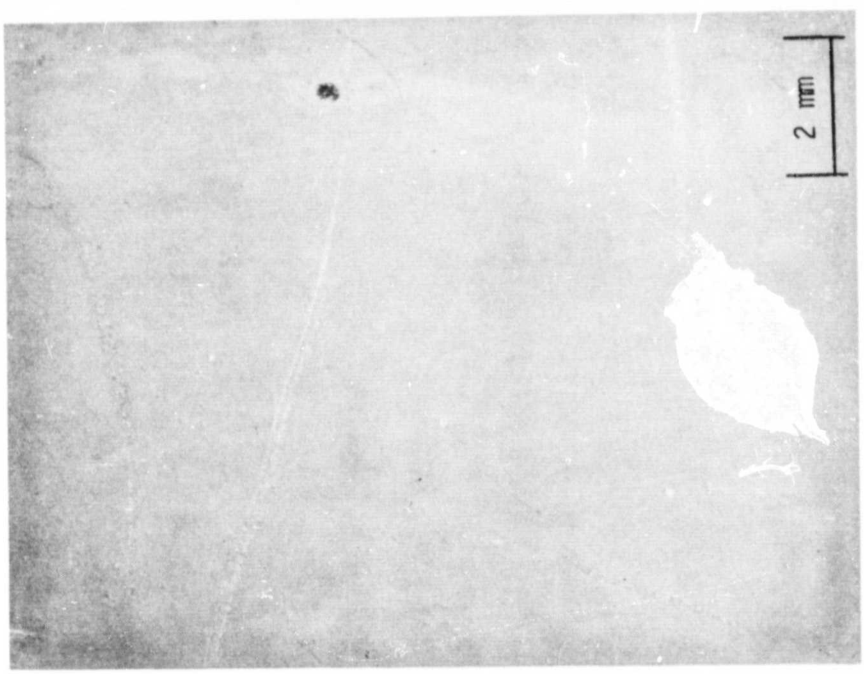


q) 7554, Liquid

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (~10X)



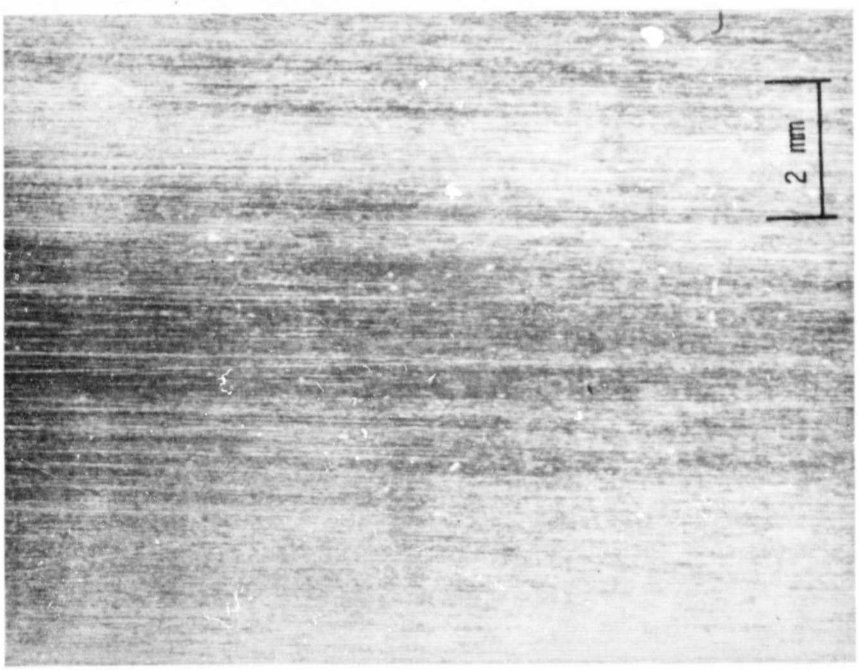
r) 7556, Vapor



s) 7556, Liquid

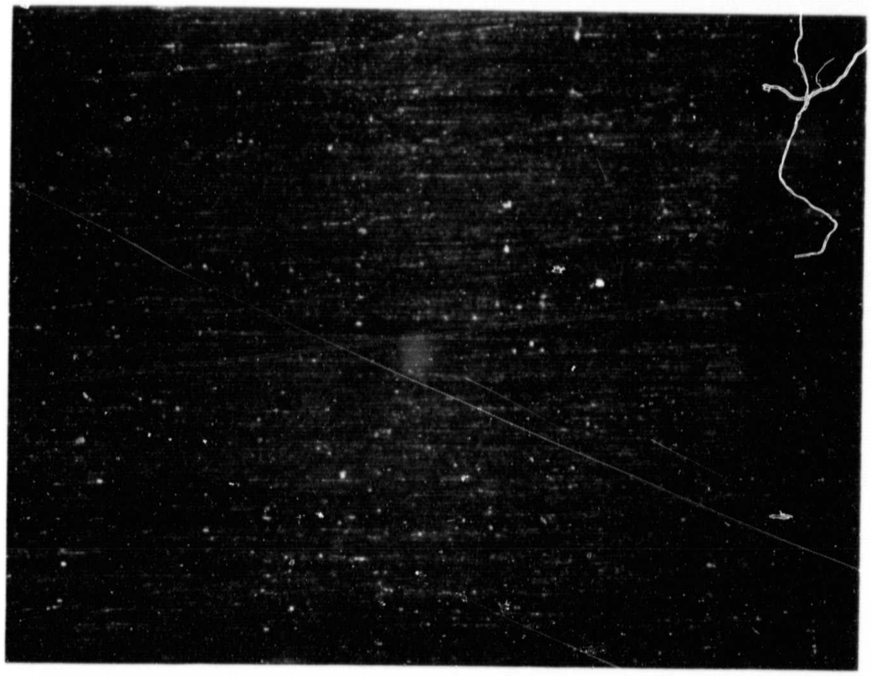
Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (∿10X)

t) 7573, Vapor

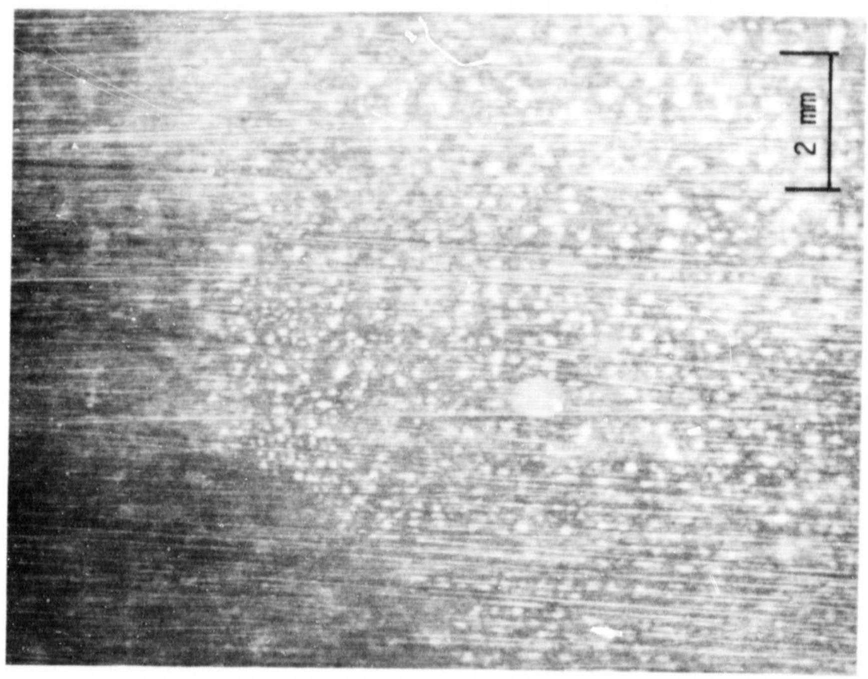


u) 7573, Liquid

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (∿10X)



v) 7574, Vapor



w) 7574, Liquid

Figure G-23 (Cont'd): Low Magnification Photographs of Surface Features, 39-Month Immersion (∿10X)

TABLE G-I. PIT DEPTH FREQUENCY DISTRIBUTION 39-MONTH IMMERSION COUPONS

Specimen No.	Percent of Pits in Various Depth Ranges (10^{-4} cm)									
	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10
Range	0.01-0.64	0.65-1.28	1.29-1.92	1.93-2.56	2.57-3.20	3.21-3.84	3.85-4.48	4.49-5.12	5.13-5.76	5.77-6.40
Mean	(0.325)	(0.965)	(1.61)	(2.24)	(2.89)	(3.53)	(4.17)	(4.81)	(5.61)	(6.09)
7504-V	0	44.0	32.0	24.0	0	0	0	0	0	0
7524-V	0	4.3	4.3	4.3	13.0	17.4	4.3	8.7	4.3	4.3
7556-V	5.9	52.9	23.5	11.8	0	5.9	0	0	0	0
7574-V	0	5.0	10.0	30.0	5.0	10.0	15.0	5.0	0	0
7504-L	0	19.4	22.6	38.7	9.7	9.7	0	0	0	0
7524-L	0	0	0	29.2	8.3	20.8	4.2	16.7	16.7	0
7556-L	0	85.3	7.1	7.1	0	0	0	0	0	0
7574-L	0	0	0	18.2	0	13.6	9.1	13.6	4.5	0

Specimen No.	Percent of Pits in Various Depth Ranges (10^{-4} cm)									
	Group 11	Group 12	Group 13	Group 14	Group 15	Group 16	Group 17	Group 18	Group 19	Group 20
Range	6.41-7.04	7.05.7.68	6.69-8.32	8.33-8.96	8.97-9.60	9.61-10-24	10.25-10.88	10.89-11.52	11.53-12.16	>12.16
Mean	(6.37)	(7.37)	(8.01)	(8.65)	(9.29)	(9.93)	(10.57)	(11.21)	(11.85)	
7504-V	0	0	0	0	0	0	0	0	0	0
7524-V	8.7	0	4.3	8.7	0	8.7	0	0	0	4.3
7556-V	0	0	0	0	0	0	0	0	0	0
7574-V	0	10	0	0	0	5	0	0	0	5
7504-L	0	0	0	0	0	0	0	0	0	0
7524-L	0	0	0	4.2	0	0	0	0	0	0
7556-L	0	0	0	0	0	0	0	0	0	0
7574-L	0	18.2	4.5	4.5	0	9.1	0	4.5	0	4.5

TABLE G-II. PIT AREA FREQUENCY DISTRIBUTION: 39-MONTH IMMERSION COUPONS

Specimen No.	Percent of Pits in Various Area Ranges 10^{-7} cm (Average)										
	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10	Group 11
Range	0.001-0.049	0.050-0.099	0.100-0.490	0.500-0.990	1.000-4.999	5.000-9.999	10.000-14.999	15.000-19.999	20.000-24.999	25.000-29.999	30.000-34.999
Mean	(0.025)	(0.0745)	(0.295)	(0.0745)	(3.000)	(7.500)	(12.000)	(17.500)	(22.500)	(27.500)	(32.500)
7504-V	0	0	8.0	8.0	36.0	8.0	12.0	8.0	6.7	8.0	4.0
7524-V	0	0	0	0	4.3	4.3	4.3	0	0	0	13.0
7556-V	0	0	0	0	17.6	17.6	5.9	11.8	11.8	5.9	11.8
7574-V	0	0	0	0	5.0	0	5.0	10.0	5.0	5.0	5.0
7504-L	0	0	9.7	9.7	22.6	22.6	3.2	6.5	0	0	3.2
7524-L	0	0	0	0	0	0	0	0	4.2	8.3	8.3
7556-L	0	0	14.3	42.9	35.7	0	0	0	0	0	0
7574-L	0	0	0	9.1	0	0	4.5	0	0	4.5	4.5

Specimen No.	Percent of Pits in Various Area Ranges 10^{-7} cm (Average)										
	Group 12	Group 13	Group 14	Group 15	Group 16	Group 17	Group 18	Group 19	Group 20	Group 21	Group 22
Range	35.000-39.999	40.000-44.999	45.000-49.000	50.000-54.999	25.000-79.999	80.000-104.999	105.000-129.999	130.000-154.999	155.000-179.999	180.000-204.999	105.000-229.999
Mean	(37.500)	(42.500)	(47.500)	(52.500)	(67.500)	(92.500)	(117.500)	(142.500)	(167.500)	(192.500)	(217.500)
7504-V	0	0	0	0	0	0	0	0	0	0	0
7524-V	8.7	0	0	13.0	9.1	22.0	17.0	0	0	0	0
7556-V	5.9	0	0	11.8	0	0	0	0	0	0	0
7574-V	5.0	0	0	10.0	10.0	5.0	0	0	0	0	0
7504-L	0	3.2	0	6.5	3.0	0	3.0	0	3.0	0	0
7524-L	8.3	4.2	0	25.0	8.3	4.2	0	4.2	4.2	4.2	0
7556-L	0	0	0	7.1	0	0	0	0	0	0	0
7574-L	4.5	0	0	13.6	13.6	0	0	9.1	4.5	4.5	4.5

Specimen No.	Percent of Pits in Various Area Ranges 10^{-7} cm (Average)										
	Group 23	Group 24	Group 25	Group 26	Group 27	Group 28	Group 29	Group 30	Group 31	Group 32	Group 33
Range	230.000-254.999	255.000-279.999	280.000-304.999	305.100-404.999	405.000-504.999	505.000-604.999	605.000-704.999	705.000-804.999	805.000-904.999	905.000-1004.999	>1005.000
Mean	(242.500)	(267.500)	(292.500)	(355.000)	(455.000)	(555.000)	(655.000)	(755.000)	(855.000)	(955.000)	
7504-V	0	0	0	0	4	0	0	0	0	0	0
7524-V	0	0	4.0	0	0	0	0	0	0	0	0
7556-V	0	0	0	0	0	0	0	0	0	0	0
7574-V	0	0	10.0	4.0	4.0	0	4.0	0	4.0	0	4.0
7504-L	0	0	0	3.0	0	0	0	0	0	0	0
7524-L	8.3	4.2	0	0	0	0	0	0	0	0	0
7556-L	0	0	0	0	0	0	0	0	0	0	0
7574-L	0	0	9.1	9.1	4.5	4.5	0	0	0	0	0

TABLE G-III: PIT DEPTH/DIAMETER FREQUENCY DISTRIBUTION: 39-MONTH IMMERSION COUPONS

Specimen No.	Percent of Pits with Various Diameter/Depth (1/d) Ratios							
	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
Ratio Range Mean	0.01-0.20 (0.105)	0.21-0.41 (0.31)	0.42-0.62 (0.52)	0.63-0.83 (0.73)	0.84-1.04 (0.94)	1.05-1.25 (0.94)	1.26-2.00 (1.63)	>2.00
7504-L	48.4	25.8	12.9	6.50	0	3.20	3.20	0
7504-V	52.0	40.0	4.0	4.0	0	0	0	0
7524-L	91.7	8.3	0	0	0	0	0	0
7524-V	61.0	30.4	4.3	4.3	0	0	0	0
7556-L	28.6	50.0	14.3	0	0	7.1	0	0
7556-V	88.2	11.8	0	0	0	0	0	0
7574-L	63.6	27.3	0	4.5	0	0	0	0
7574-V	90.0	10.0	0	0	0	0	0	0